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ARMY-APPROVED SAFE MATERIALS TO REPLACE DOP IN MASK AND FILTER TESTING: EMERY 3002 (ETHYLFLO 162) AND EMERY 3004 (ETHYLFLO 164)



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RESEARCH AND TECHNOLOGY DIRECTORATE

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In April 1986, the U.S. Army's Office of The Surgeon General (OTSG) took the position that dioctyl phthalate (DOP) was a suspected carcinogen. The U.S. Army had used DOP for many decades in nondestructive servicability testing of respirator canisters and protective filters, and in a variety of aerosol penetration studies including mask leakage and face fit. A program was initiated in September 1987 to find a safe replacement material for DOP, under sponsorship of the Product Assurance Directorate (PAD), CRDEC. A synthetic lubricant named "Emery 3004," from the class of compounds called poly-alpha olefins (PAOs), was approved by the OTSG on 8 January 1992 for use Army-wide as a safe replacement for DOP in "hot smoke" and "cold smoke" testing machines. Another less-viscous PAO, "Emery 3002," was also approved by the OTSG on 24 February 1993 for Army-wide use in cold-smoke applications. The Ethyl Corporation (Boca Raton, LA) is the primary manufacturer of these PAOs, under the trade names "Ethylflo 162" (repackaged as Emery 3002), and "Ethylflo 164" (repackaged as Emery 3004). These materials are extremely useful DOP replacements. They perform at least as well as DOP in various testing machines. They can replace DOP directly in existing machines without modification, and are recommended to replace DOP as soon as is practicable.

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PREFACE

The work described in this report was authorized under Sales Order No. 3J9HO8. This work was started in February 1992 and completed in June 1993.

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ARMY-APPROVED SAFE MATERIALS TO REPLACE DOP IN MASK AND FILTER TESTING:

EMERY 3002 (ETHYLFLO 162) AND EMERY 3004 (ETHYLFLO 164)

1. INTRODUCTION

1.1 Background

Di (2-ethylhexyl) phthalate, also called dioctyl phthalate, di-sec octyl phthalate, DOP, or DEHP, is a widely used industrial material. Over ninety percent of the material produced is used as a plasticizer, primarily for PVC plastics. The properties of DOP that make it useful as a plasticizer, including low vapor pressure, chemical stability, and insolubility in water, also make it useful as a test aerosol. DOP aerosols are used in respirator fit testing, HEPA filter testing, aerosol research, aerosol instrument calibration, and other applications. These uses involve human occupational exposure to submicrometer-sized DOP aerosols, often briefly but in moderately high concentrations. 1

The U.S. Army routinely performs 100% quality control testing of filter canisters manufactured for use with field-issue gas masks, and periodic sampling and testing of canisters stored in its supply depots. In April, 1986, the U.S. Army's Office of the Surgeon General (OTSG) announced that DOP posed potentially serious health risks to workers, and placed severe restrictions upon testing with it; agencies were also informed that dioctyl sebacate (DOS) would no longer be acceptable as a DOP replacement material, and that similar restrictions would apply for both. These restrictions included occupational exposure monitoring of workers exposed to DOP aerosols and liquid, medical surveillance, issue of personal protective equipment, formal notification to workers of associated risks, and labeling of work areas as "cancer suspect agent areas."

1.2 Summary of the Successful DOP Replacement Program

The above actions placed severe restrictions upon routine, 100% quality assurance testing of filters and other equipment. For this reason, in 1987 the U.S. Army initiated a detailed study of the problem of finding an acceptable substitute material for DOP that could meet all standard military test specifications while itself being a non-carcinogen and, ideally, having other attributes including acceptable acute inhalation toxicity, low cost, ready availability, and the ability to replace DOP directly in machines at test installations without retrofit or other modification of these machines.

The Army had used DOP for many decades in non-destructive servicability testing of respirator canisters and protective filters, and in a variety of aerosol penetration studies including mask leakage and face fit. The program initiated in September 1987 to find a safe replacement material for DOP was sponsored by the Product Assurance Directorate (PAD).^{2.3} A synthetic lubricant named "Emery 3004," from the class of compounds called poly-alpha olefins (PAOs), was approved by the OTSG on 8 January 1992 for use Army-wide as a safe replacement for DOP in "hot smoke" and "cold smoke" testing machines. Emery 3004 was approved after successfully passing three tiers of mutagenicity testing that included the Ames system assay, the sex-linked recessive lethal test in fruit flies, and the rodent bone marrow micronucleus assay performed with rats.

A less-viscous PAO, "Emery 3002," also was also approved by the OTSG on 24 February 1993 for Army-wide use in cold-smoke applications (Appendix A). The Ethyl Corporation⁴ is the primary manufacturer of these PAOs, under the trade names "Ethylflo 162" (repackaged as Emery 3002), and "Ethylflo 164" (repackaged as Emery 3004). These materials are extremely useful DOP replacements. They perform at least as well as DOP in various testing machines. They can replace DOP directly in existing machines without modification. They are inexpensive, readily available, and should continue to remain so in the future. Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are readily specifiable, non-corrosive, free of natural impurities, thermally and chemically stable, and safe to work with. They are recommended to replace DOP in Army-wide testing as soon as is practicable.

2. PROPERTIES OF THE ARMY-APPROVED MATERIALS

2.1 General

With two safe DOP replacement materials now approved by the OTSG for Armywide use, a choice is available between them for specific applications. As will be seen, Emery 3004/Ethylflo 164 works very well in both "hot smoke" and "cold smoke" machines, while Emery 3002/Ethylflo 162 is recommended only for cold smokes or machines operating at moderate temperatures. This is because 3004/164 has a flash point of 225°C, well above typical "hot pot" operating temperatures of 17C-180°C, while 3002/162 has a flash point of only 164°C. Auto-ignition occurs in either material only at much higher temperatures; nevertheless, we recommend that only 3004/164 be used in "hot smoke" machines.

The PAOs have many unique and interesting properties. They represent a family of synthetic lubricating oils that were unknown a few decades ago. Yet they have replaced natural petroleum products in a great variety of applications, primarily because they work better, they are purer, and they are inexpensive.

Material Safety Data Sheets (MSDS) for Ethylflo 162/Emery~3002 and Ethylflo 164/Emery~3004 are presented in <u>Appendixes B and C</u>, respectively. Manufacturer's specifications for both products (and some other PAOs) are given in Appendix D.

2.2 Molecular Structure

Because Ethyl Corp. (and thus Emery) PAOs are produced by combining 10-carbon decene molecules, the resulting products do not contain distributions of sequential carbon chain lengths, like those found in common petro-

leum products, which are difficult to separate by distillation. Instead, each PAO consists of chain lengths in multiples of 10 which, in principle, should allow easier distillation to obtain samples of a single kind of molecule of high purity. Approximate analyses for the PAOs of interest here are listed in Table 1. The last digit of the "3000" or "160" numbering scheme indicates the viscosity in centistokes of that particular mixture at 100°C.

Table 1. Approximate Analyses by Carbon Chain Length for Emery 3002/Ethylflo 162 and Emery 3004/Ethylflo 164.

Ethyl/ Emery		Percer	itages by Ca	rbon Chain	Length	
Product	C20	C30	C40	_C50_	C60	<u>C70</u>
162/3002	97-99	1.0				
164/3004	0.60	82.1	16.9	1.0	2.0	

Table 1 shows that Ethylflo 164/Emery 3004 is a mixture of of C_{30} and C_{40} polyolefins with molecular weights (MW) of 420 and 560, respectively, and traces of other carbon chain lengths. But the Ethylflo 162/Emery 3002 is nearly all "dimer," i.e., 20-carbon chains each formed from the combination of two decene-1 (10 carbon) molecules.

The molecules comprising the PAOs become progressively non-linear with increasing size. Thus, for larger sizes, the molecules increasingly become isomers of one another. The manufacturing process begins with decene-1, a linear hydrocarbon molecule of 10 carbons with one double bond at the first position. The decene-1 is polymerized in the presence of a BF3 catalyst, hydrogenated to saturate any remaining double bonds, and distilled into fractions (dimer C_{20} , trimer C_{30} , quatrimer C_{40} , ...). These are blended to produce desired product mixtures for various applications, as indicated in Table 1. There are many other PAOs of higher viscosities, which are not shown.

Chemists generally agree that the C_{20} dimer is nearly straight-chained but, because polymerization occurs at the site of the decene-1 double bond, a "bending" occurs such that the dimer molecule has the form:

Structural theories of the PAOs suggest that as the molecules become larger chain attachment occurs at selected sites such that the C_{30} trimer has the form:

while the C_{40} quatrimer has a form resembling:

Materials other than decene-1 can be used to make PAOs, but decene-1 gives products with the most desirable range of viscosities for lubricants. To prevent discoloration due to oxidation catalyzed by contact with metals during us antioxidants can be added to PAOs in concentrations well below one percent.

2.3 Physical Properties

Some physical properties of the poly-alpha olefins (PAOs) discussed here are given in $\underline{\text{Table 2}}$. Many others are given in Appendix D, and also in Appendixes B and C.

Table 2. Properties of Poly-Alpha Olefins (PAOs).

Ethyl/ Emery Product	Pour Point, ^O C	Flash Point, ^O C	Fire Point, ^O C	Auto-Ignition Point, ^O C	Specific Gravity
162/3002	-65	164	178	324	0.80
164/3004	-69	225	250	343	0.82

The viscosities, in centistokes (cSt), of Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are plotted versus Centigrade temperature in <u>Figure 1</u>. These data were obtained from manufacturer's specification sheets in Appendix D. At 100° C, the viscosity of 162/3002 is 1.80 cSt, approximating the value "2" indicated by the last digits of the product numbers. For 164/3004 at 100° C, the viscosity is 3.90 cSt, approximating "4" in the product numbers.

 $\underline{\text{Table 3}}$ contains manufacturer's 4 viscosity data for the Ethylflo/Emery PAOs, as well as values measured at ERDEC over a limited temperature range for dioctyl phthalate (DOP).

Temperature, OC	Viscosity, Centistokes (cSt)					
	Ethylflo 162/Emery 3002	Ethylflo 164/Emery 3004	DOP			
-40	310.0	2460.0				
-18	62.0	341.0				
15			109.80			
25			58.36			
35			33.90			
40	5.54	16.8				
50			17.55			
100	1.80	3.90				

Table 3. Viscosities of PAOs and DOP.

Viscosity is an important parameter when liquids are sprayed through two-fluid atomizers to produce fine aerosols such as those used in a variety of laboratory research applications. As temperature is reduced, the ratio of viscosities of 164/162 or 3004/3002 increases. At 100° C this ratio is $u_4/u_2 = 3.90/1.80 = 2.17$. At 40° C it is 16.8/5.54 = 3.03, and at -18° C the ratio is 341/62.0 = 5.50 (see Appendix D).

Thus, at lower temperatures, the viscosity of Ethylflo 162/Emery 3002 increases more slowly than that of Ethylflo 164/Emery 3004. If room temperature is taken as 20°C, the viscosity ratio (Figure 1) becomes approximately 35/10 = 3.5. When "cold smokes" are generated by spraying at room temperature, these numbers indicate that 162/3002, being much less viscous than 164/3004, should "break up" more readily to form the smaller droplets of the two liquids. Laskin nozzles, which use pressurized gas injected through jets beneath the surface of the liquid being aerosolized, represent a more complex case.

Another important parameter in liquid breakup through nozzles is surface tension (ST). Values of ST over a limited temperature range were measured for the PAOs and for DOP at ERDEC, with the results shown in <u>Table 4</u>.

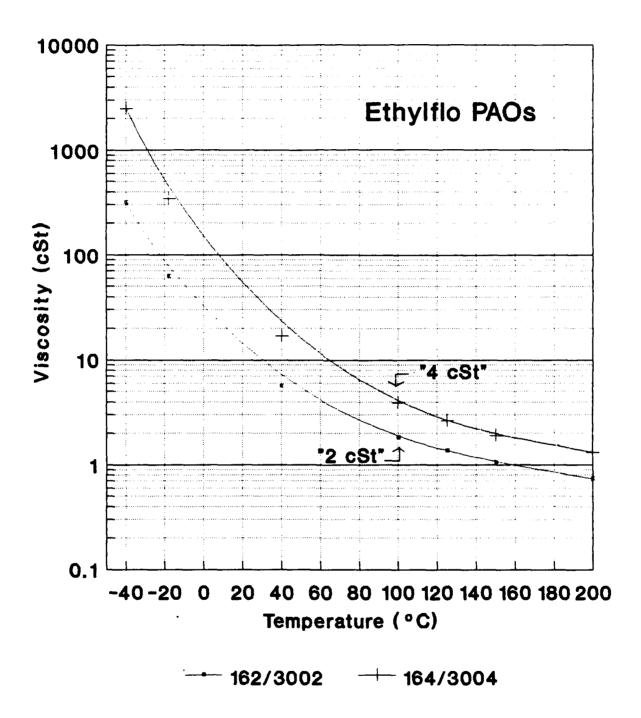


Figure 1. Viscosity (cSt) of PAOs Versus Temperature (°C).

Table 4. Surface Tensions* of PAOs and DOP.

	Surface Tension (dynes/cm)					
Temperature, OC	Ethylflo 162/Emery 3002	Ethylflo 164/Emery 3004	DOP			
15	28.8	29.9	32.1			
25	28.0	29.0	31.3			
35	27.1	28.2	30.5			
50	26.0	27.0	29.2			

^{*} Measured at ERDEC.

The data presented in this report for smokes produced from Ethylflo 162/Emery 3002 and Ethylflo 164/Emery 3004 are experimental. Some progress has been made in the prediction of droplet size distributions from spray nozzles. A well-known equation for liquid breakup was published in 1939 by Nukiyama and Tanasawa⁵, but this was deficient in that it contains no factors relating to nozzle design. Also, except for sonic flow, there is no dependence upon the physical properties of the gas which is used to disperse the liquid. These factors were pointed out by Gretzinger and Marshall⁶, who proved their importance.

An empirical equation which answers some of the objections of the Nukiyama-Tanasawa expression, and which appears to fit experimental data more closely, has been given by Kim⁷ for convergent type atomizers. The mixture of English and metric units is Kim's:

$$\overline{x}_{m} = \frac{249}{A^{0.365} v_{rel}^{1.144}} \left(\frac{\sigma_{1}}{\rho_{a}}\right)^{0.572} \left(\frac{\mu_{1}^{2}}{\sigma_{1}\rho_{1}}\right)^{0.161} + \frac{1260}{v_{rel}^{0.54}} \left(\frac{\mu_{1}^{2}}{\sigma_{1}\rho_{1}}\right)^{0.17} \left(\frac{M_{1}}{M_{a}}\right)^{m}$$
(4)

where

 \bar{x}_{m} = mass mean diameter (microns)

 μ_1 = liquid viscosity (centipoises)

 $\rho_1 = \text{liquid density (lb/ft}^3$)

 $\rho_a = gas density (1b/ft^3)$

 σ_1 = liquid surface tension (dynes/cm)

v_{rel} = difference in gas and liquid velocity (ft/sec)

A = flow area for the gas (in.²)

 M_a and M_1 = mass flow rates for gas and liquid respectively

m = 1 for $M_1/M_A > 1/3$ and m = 0.5 for $M_1/M_A < 1/3$

At large gas-to-liquid flow rates, a limiting small particle size is achieved and the value for this particle size is given by the first term on the right side of the equation. At small gas-to-liquid flow rates, particle size will be appreciably larger and controlled primarily by the second term on the right of the equation. Significantly, factors are present which include the design of the nozzle, namely, its area, as well as the density of the gas stream.

It can be seen in <u>Equation 4</u> that liquid viscosity and surface tension are both well represented. Droplet diameter increases as the 0.572 power of surface tension. Offsetting this are the terms in parentheses taken to the small powers 0.161-0.17, where the square of viscosity is divided by liquid surface tension and density. Thus at face value, the equation appears to support the view that increasing either viscosity or surface tension, or both, will tend to give larger droplet size distributions from sprayers.

3. TYPICAL PERFORMANCE DATA

3.1 General

There are two general types of smoke generators: those for "hot" smokes used in mask and filter testing, and those for "cold" smokes used in filter testing as well as for tests such as mask face fit or leakage other than through the filter canister.

Often a hot smoke is generated by vaporization/recondensation of a liquid, particularly when a "monodispersed" aerosol is required by test directives. Monodispersity implies a very narrow droplet size distribution. For example, U.S. Army mask canisters must be challenged by smokes of 0.2-0.3 um mean droplet diameter, with a geometric standard deviation (GSD) equal to or less than 1.3. Examples of typical hot smoke data are contained in Appendix E.

There are other designs for hot smoke generators. For example, the Los Alamos National Laboratory (LANL) has built a prototype system² called "LAMAPP," an acronym for Los Alamos Monodispersed Aerosol Prototype Penetrometer. This system uses a cold pot to hold the test liquid, which is finely aerosolized by a Laskin nozzle. The aerosol then passes through a heated column and into a mixing chamber. The droplets are vaporized, and recondensation then occurs on very tiny salt (NaCl) nuclei that are much smaller than the resulting, monodispersed, smoke droplets. In our tests, Ethylflo 162/Emery 3002 was found to be the best-performing of all liquids in the LAMAPP system. But in the standard TDA-100 hot pot, hot smoke machine (Appendix E and Reference 2), the best performer was Ethylflo 164/Emery 3004. Due to the vaporization and recondensation temperatures, 162/3002 works poorly in the TDA-100.

Both materials, 162/3002 and 164/3004, can be used in cold smoke applications, and both have attained full approval for use by the U.S. Army's Office of the Surgeon General (OTSG). Thus the choice of one or the other for a given application will include consideration of factors such as operating temperature, size distributions and concentrations obtained from various nozzles, and parameters including flow rates, pressures, and liquid/gas mass flow ratios.

The one restriction has been noted earlier and can be rephrased here: Ethylflo 162/Emery 3002 should not be used in hot pot machines (above 100°C).

3.2 <u>Hot Smoke (Vaporization/Recondensation) Applications</u>

Until the recent approval from the OTSG of Ethylflo 162/Emery 3002 (Appendix A) for cold smoke testing, the authors had concentrated their efforts upon Ethylflo 164/Emery 3004, which had OTSG approval a year earlier, for both hot and cold smoke applications. Thus, many existing data were taken using 164/3004 to generate cold smokes, and some of these data are presented in Appendix F. 164/3004 was intended primarily for hot smoke generation against stringent military specifications. Some data of this kind are included in Appendix E.

Great success in both hot and cold smoke testing with Emery 3004/ Ethylflo 164 has been achieved at the Hanford (WA) Nuclear Site, which is now operated by the Westinghouse Hanford Company (WHC) for the U.S. Department of Energy (DOE). In an October 1992 report, which is reproduced here in full (Appendix G), WHC scientists reported that their own test data confirmed ERDEC's conclusions that 3004/164 performs at least as well as DOP, and somewhat better than DOS (dioctyl sebacate, another replacement material), in a variety of machines.

WHC had been using DOS in their Q-76, Q-107, and Q-127 (TDA-100) testers because the use of DOP had been curtailed due to its possible carcinogenicity. DOS was causing gumming and other fouling problems not unlike those encountered with corn oil in other machines. DOE was dealing with the problem of deciding whether to convert to a suitable DOP substitute such as one of the authors' materials, or to replace their machines with new units that incorporate completely different "cold smoke" technology. These would generate smokes with much poorer "monodispersity," i.e., wider particle size distributions, than currently are achievable using hot smoke technology.

WHC reported (Appendix G) that "Emery 3004 has several advantages over approved performance testing chemicals, including that it is not considered a carcinogen or suspect carcinogen; therefore, respiratory protection is not required during testing.' 'Additionally, Emery 3004 does not cause buildup on or plugging of the test equipment like DOP or DOS.' 'By reducing the maintenance required on equipment, use of Emery 3004 increases the efficiency of Westinghouse Hanford Company operations." Hazardous waste is greatly reduced using 3004/164 with nuclear dust filters. If DOP is used, the waste is classed as both nuclear and carcinogen contaminated; disposal is much more complex.

As a result of this work, DOE Headquarters has notified WHC that they have approved Emery 3004/Ethylflo 164 as a challenge aerosol for the in-place testing of high-efficiency particulate air filter systems, effective September 1992. This should lead swiftly to approval for similar use throughout DOE.

The TDA-100 (Q-127) hot-smoke monodispersed aerosol penetrometer is described in Appendix E. This is a standard test machine, used worldwide. Some typical data printouts from a laser aerosol spectrometer are shown in Appendix E, comparing specifications measured for DOP with those for Emery 3004 (Ethylflo 164).

The DOP data indicate an aerosol with a geometric mean diameter (GMD) of 0.2004 um and a geometric standard deviation (GSD) of 1.262, well within the

1.30 specification for DOP hot test smokes. The "Emery 3004" smoke has a GMD of 0.2083 um (much like DOP), and a GSD of 1.245. In this example, both DOP and its replacement material behave very much alike.

In order to replace DOP with Emery $3004/\Xi$ thylflo 164 in a hot smoke penetrometer machine such as the TDA-100, only minor adjustments of the machine's operating controls are required². For example, with a "hot pot" temperature of 172° C, DOP will produce smokes having GMDs over the range 0.19-0.25 um and GSDs of 1.21-1.54, giving nephelometer ("Owl") readings of 29-48 degrees, particle size control settings, "V," of 38-100 volts, and quench air to vapor flow rates, "Q," of 80/20. Emery 3004 at $170-180^{\circ}$ C gives GMDs of 0.20-0.30 um and GSD = 1.23 for Owl readings of 43-50 degrees, V = 46-75, and Q = 90/10.

These data and many additional data taken by independent investigators confirm that Ethylflo 164/Emery 3004 performs extremely well in hot smoke applications^{2,3}. It possesses virtually every attribute that could be desired in a direct replacement material for DOP and DOS. It is also very effective in cold smoke applications, as will be discussed below.

Ethylflo 162/Emery 3002 outperforms 164/3004 only in the "hybrid" of hot smoke applications mentioned earlier: the LAMAPP machine. In the LAMAPP, because the liquid is first aerosolized using Laskin nozzles in a pot at room temperature, the 162/3002 gives a fine spray of liquid droplets which are then evaporated to allow recondensation upon tiny salt nuclei.

3.3 <u>Cold Smoke (Room Temperature) Applications</u>

Some examples of cold smoke data obtained using Emery 3004/Ethylflo 164 and Emery 3002/Ethylflo 162 are contained in Appendix F. As has been mentioned, 3004/164 has been more extensively tested in cold smoke applications than has 3002/162, since the former was approved by the U.S. Army's OTSG a year earlier than the latter.

A cold smoke machine that is receiving wide acceptance is the TSI, Inc., Model 8110 Automated Filter Tester (AFT), which is described briefly in Appendix F. The tester generates an aerosol which is used to challenge a test filter, and filter penetration is calculated by a microprocessor. Once a filter or filter canister is installed in the holder, two buttons are pushed and the test runs automatically with results displayed at the conclusion. The AFT has a "low" and a "high" aerosol concentration mode, enabling challenge aerosols to be generated covering the range 15-100 mg/m³.

The Model 8110 was used in collaborative measurements involving scientists from what is now ERDEC, and the National Institute for Occupational Safety and Health (NIOSH) in Morgantown, WV. Data are tabulated in Appendix F for DOP, Emery 3004 (Ethylflo 164), and two other candidate DOP replacement materials. Aerosols were analyzed using the TSI Model 3071 Electrostatic Classifier (EC) with the TSI Model 3020 Condensation Nucleus Counter (CNC). Data plots showed that the performances of DOP and Emery 3004/Ethylflo 164 were very similar.

Also used was the TSI Model 3932 Differential Mobility Particle Sizing System DMPS), which prints a bar chart of particle size distribution versus mobility channel, as shown in Appendix F. Emery 3004/Ethylflo 164 was found to

give an aerosol mass concentration from the Model 8110 that was greater than the DOP yield by 52 percent in the "high" mode and 60 percent in the "low" mode.

Other tests were conducted in the Edgewood Area of Aberdeen Proving Ground using a new prototype machine developed by the Los Alamos National Laboratory (LANL) which is variously known as the High Flow Alternative Test System, HIFATS, HFATS, or simply ATS. This system is described briefly in Appendix F. Aerosol is generated in a cold pot through a selectable number of Laskin nozzles (to control mass concentration), and is analyzed before (with dilution) and after filter penetration using a Laser Aerosol Spectrometer (LAS). Tests were run with Emersol 875 (isostearic acid, another candidate DOP replacement material), and with Emery 3004/Ethylflo 164 at 1500 CFM air flow rate under various conditions.

The HFATS operates on a new principle in that a broad range of particle (droplet) sizes is generated, but only those sizes ranges that are of interest need to be monitored to determine filter penetration. The system does, however, print out the full range penetrations in each size interval or "bin."

Thus, the first of three HFATS data tables for Emery 3004/Ethylflo 164 in Appendix F shows droplet diameters ranging from 0.075 um in "Bin 0" to 0.363 um in "Bin 12." But it is in "Bin 5" where the greatest filter penetration occurs, corresponding to a challenge diameter of 0.209 um. This is because most filters exhibit maximum penetration (minimum protection) at a particle size where two filtration mechanisms overlap: mechanical blocking and convection to surfaces at larger particle sizes; electrostatic attraction at smaller sizes.

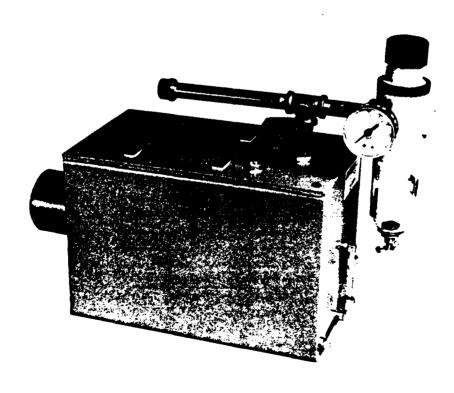


Figure 2. Air Techniques, Inc., Model TDA 4-A Aerosol Generator.

The final cold smoke generator described briefly in Appendix F is the ATI, Inc., Model TDA-4A, shown in <u>Figure 2</u>. This unit features a total of eight Laskin nozzles, which are selectable by opening or closing a pair of valves so that, e.g., one, three, six, or eight nozzles can be used. The liquid to be aerosolized in placed in the bottom of the housing, submerging the nozzle outlets. Compressed air creates the liquid aerosol by shearing the liquid.

Most cold smoke generators use some configuration of Laskin nozzles, which produce rather wide particle size distributions. To limit the upper size range, thus conserving most of the liquid which is sprayed in droplets too large to be useful for test purposes, some sort of crude pre-sizing is used. All of the generators discussed in this section use some system of pads, filters, impactors, or baffles to limit mean particle sizes of their output aerosols to values typically near 0.7 um.

These resulting size distributions are still much broader than those from hot smoke, "monodispersed" aerosol machines, which produce aerosols having geometric standard deviations (GSDs) of 1.30 or less, and which can be smaller than 1.20 in well-tuned machines. By contrast, cold smoke machines typically produce aerosols with GSDs of 1.60 - 1.90, or even larger values.

Experiments were conducted in our laboratory, using the Model TDA-4A cold smoke aerosol generator, to allow direct comparisons of the performances of Emery 3002/Ethylflo 162, Emery 3004/Ethylflo 164, and DOP with each other in true cold smoke applications. We have shown³ that 3004/164 is clearly superior in hot smoke, monodispersed machines, and that 3002/162 works best in the hybrid (cold pot, hot smoke) LAMAPP machine.

The TDA-4A output was connected through a pair of aerosol diluters to a TSI, Inc., Differential Mobility Particle Sizer (DMPS) with a Condensation Nucleus Counter (CNC). Air pressure was 20 psi. The liquids tested were DOP, Ethylflo 162/Emery 3002, and Ethylflo 164/Emery 3004. One Laskin nozzle was operated to limit TDA-4A output and conserve liquids. The air flow from one nozzle at 20 psi was 0.391 liters per minute. Mass concentrations and other data for the three liquids are shown in Table 5.

Table 5. Data for Three Liquids in the Model TDA-4A Aerosol Generator, Operating with One Nozzle at 20 psi Pressurization.

	Mass Concentration,	Geometric	Mean Droplet Di	ameter, um
Liquid	g/m ³	Count	Surface	Volume
Ethylflo 162/Emery 3002	5.03	0.202	0.287	0.333
Ethylflo 164/Emery 3004	5.61	0.374	0.482	0.532
Dioctyl Phthalate (DOP)	4.79	0.384	0.503	0.548

Before looking at the droplet size distributions for aerosols of these three liquids, some interesting observations can be made concerning the data in Table 5. First, note that the mass concentration yield for DOP is the smallest

TSI DIFFERENTIAL MOBILITY PARTICLE SIZER

ETHYLFLO 162

SAMPLE # 1 AEROSOL FLOW RATE: .3 LPM

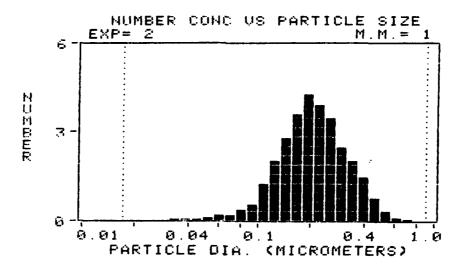
DATE: 07/23/93

MAXIMUM DIA. MEASURED: .886 UM

MINIMUM DIA. MEASURED: .017 UM E

START: 11:03:36 END: 11:33:37

MEAS. MODE: 1



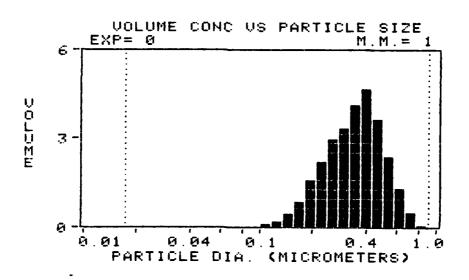


Figure 3. DMPS Particle Size Distributions for Ethylflo 162/Emery 3002.

TSI DIFFERENTIAL MOBILITY PARTICLE SIZER

ETHYLFLO 16#

SAMPLE # 1

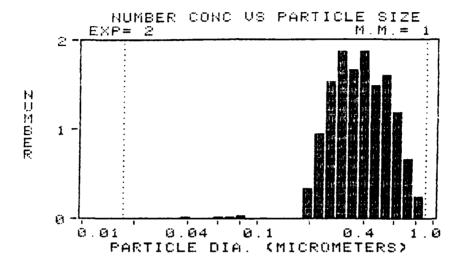
AEROSOL FLOW RATE: .3 LPM MAXIMUM DIA. MEASURED: .886 UM

MEAS. MODE: 1 START: 14:51:21

DATE: 07/20/93

MINIMUM DIA. MEASURED: .000 UM

END: 15:24:21



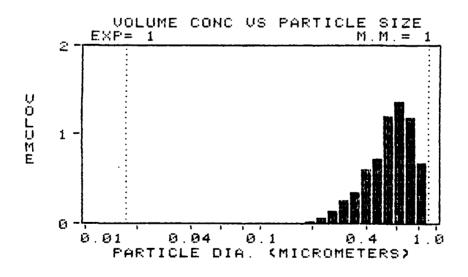


Figure 4. DMPS Particle Size Distributions for Ethylflo 164/Emery 3004.

TSI DIFFERENTIAL MOBILITY PARTICLE SIZER

DOP @ 20 PSI

SAMPLE # 1

AEROSOL FLOW RATE: .3 LPM MAXIMUM DIA. MEASURED: .886 UM

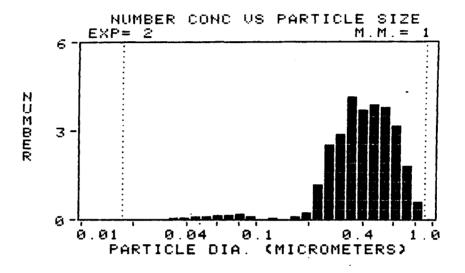
MEAS. MODE: 1 START: 09:55:29

DATE: 07/29/93

MINIMUM DIA. MEASURED: .017 UM

END: 10:22:10

minimum Dia. measured: .017 um End: 10:22:10



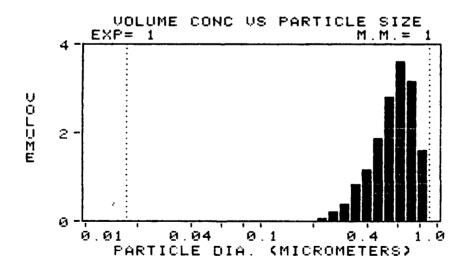


Figure 5. DMPS Particle Size Distributions for Dioctyl Phthalate (DOP).

of the three liquids. It is about 15 percent smaller than that of 164/3004. Second, it is seen that the mean droplet diameters of DOP and 164/3004 are quite similar, with the DOP droplets being the slightly larger ones. But the 162/3002 droplets are by far the smallest of any, and lie in a size range that is nearly optimum for filter penetration. Thus Ethylflo 162/Emery 3002 seems capable of producing much higher counts of more penetrating particles with similar mass flow rates or concentrations to those of Ethylflo 164/Emery 3004 and/or DOP.

Droplet size distributions and other data for aerosols of these three liquids in the TDA-4A are shown in <u>Figures 3-5</u>. Number or count distributions (top) and volume or mass distributions (bottom) are shown in each figure. In general, the number distributions are quite symmetrical, while the volume distributions are skewed toward the right. In Figure 3, both the number and volume peaks are seen to lie far to the left of those in Figures 4 and 5 for 164/3004 and DOP, respectively.

4. CONCLUSIONS

The information and discussions presented in this technical report lead to the following conclusions:

- There are now two liquids approved by the U.S. Army's Office of the Surgeon General (OTSG) for use in mask and filter testing and in general aerosol testing to replace the suspected carcinogen DOP; these are Emery 3004/Ethylflo 164 (approved by OTSG on 8 January 1992), and Emery 3002/Ethylflo 162 (approved by OTSG on 24 February 1993, see Appendix A);
- Emery 3002/Ethylflo 162 should be used only in cold pot machines since its flash point of 164 °C is close to that of temperatures used in "hot pot" machines;
- The physical and chemical properties of both liquids are well known (Tables 1-4, Figure 1, Appendixes B,C,D);
- The molecular structures of the two liquids are completely different (Table 1); Emery 3002/Ethylflo 162 is almost pure "dimer," its molecules contain 20 carbon atoms; Emery 3004/Ethylflo 162 is a mixture of mostly "trimer" (30 carbons) and some "tetramer" (40 carbons);
- Liquid breakup behavior by submerged Laskin nozzles cannot be expected to be the same as that for two-fluid pneumatic nozzles in gas or air; Equation 4 can be validated only by experiment;
- Emery 3004/Ethylflo 164 has been found to be highly useful by the Westinghouse Hanford Company (WHC, Appendix G), in many kinds of testing and in cost savings realized.

5. RECOMMENDATIONS

We recommend that:

• Emery 3004/Ethylflo 164 be used in all hot-pot, hot smoke machines that require replacements for DOP or other materials to meet safety criteria;

- Emery 3002/Ethylflo 162 be used in hybrid cold-pot, hot smoke machines such as the LANL LAMAPP (see text) to meet safety criteria;
- Emery 3004/Ethylflo 164 or Emery 3002/Ethylflo 162 be used in cold-pot, cold smoke machines such as the TSI Model 8110, the LANL HFATS, or the ATI TDA-4A, to meet safety criteria;
- Further studies are needed of the performance of both liquids in a variety of Laskin nozzle and two-fluid pneumatic nozzle systems;
- A mathematical modeling capability needs to be validated to predict droplet size distributions expected from a given nozzle under given operating parameters (e.g., Equation 4).

REFERENCES

- 1. Hinds, W.C., Macher, J.M., and First, M.W., "Size distribution of aerosols produced by the Laskin aerosol generator using substitute materials for DOP," Am. Ind. Hyg. Assoc. J. 44(7):495-500 (1983).
- 2. Carlon, H.R., Guelta, M.A., and Gerber, B.V., "A study of candidate replacement materials for DOP in filter-testing penetrometer machines."

 Technical Report CRDEC-TR-053, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD 21010-5423, March 1989.
- 3. Carlon, H.R., and Guelta, M.A., "Implementation of DOP replacement with selected materials in mask and filter testing penetrometer machines: Final report," Technical Report CRDEC-TR-370, U.S. Army Chemical Research, Development and Engineering Center, Aberdeen Proving Ground, MD 21010-5423, June 1992.
- 4. Ethyl Corporation, 451 Florida Boulevard, Boca Raton, LA 70801; TEL: (504) 388-7040, FAX: (504) 388-7848.
- 5. Nukiyama, S., and Tanasawa, Y., <u>Trans. Soc. Mech. Engrs. Japan</u> 5, 62 (1939).
- 6. Gretzinger, J., and Marshall, W.T., <u>J. A.I.Ch.E.</u> 7, 312 (1961).
- 7. Kim, K.Y., <u>Drop Size Distributions from Pneumatic Atomizers</u>, Ph.D. Thesis, University of Wisconsin (1959).

APPENDIX A

APPROVAL FROM THE U.S. ARMY SURGEON GENERAL FOR USE OF ETHYLFLO 162/EMERY 3002 IN COLD SMOKE TESTING

SGPS-PSP-E (SMCCR-CMH/3 Sep 92) (40-5e) 3rd End LTC Bratt/DSN 289-0125 SUBJECT: Request for Approval for Use of Emery 3002 Army-wide for "Cold Smoke" Spraying Systems

HQDA (SGPS-PSP), 5109 Leesburg Pike, Falls Church, VA 22041-3258
24 February 1993
FOR Commander, U.S. Army Materiel Command, ATTN: AMCSG,
5001 Eisenhower Avenue, Alexandria, VA 22333-0001

- 1. The Office of The Surgeon General (OTSG) has reviewed the previous endorsement prepared by the Army Environmental Hygiene Agency (AEHA). This office concurs with AEHA's recommendation to approve Emery 3002 for use in "cold smoke" spraying systems.
- 2. Staff point of contact for OTSG is LTC-Gary M. Bratt, DSN 289-0125. Specific technical questions should be directed to Dr. Leach, DSN 584-3980.

L RÓBERT G. CLAYPOOL

Colonel, Medical Corps

Director, Professional Services

CF:

CDR, AEHA, ATTN: HSHB-MO-T

CDR, AMC, ATTN: AMCSG-H CDR, HSC, ATTN: HSCL-P

CDR, MRDC, ATTN: SGRD-PLC

HSHB-MO-T (SMCCR-CMH/3 Sep 92) (40-5e) 2d End Dr. Leach/jls/DSN 584-3980 SUBJECT: Request for Approval for Use of Emery 3002 Army-wide for "Cold Smoke" Spraying Systems

Commander, U.S. Army Environmental Hygiene Agency, Aberdeen Proving Ground, MD 21010-5422

FOR HQDA (SGPS-PSP), 5109 Leesburg Pike, Falls Church, VA 22041-3258

- 1. Reference. Memorandum, HQDA (SGPS-PSP), Subject: Request for Approval of Emery 3004 as an Army-wide Substitute Material for Dioctyl Phthalate (DOP), 8 Jan 92.
- 2. This agency previously recommended approval of Emery 3004 as a substitute material for dioctyl phthalate in filter test apparatus. Emery 3002 is a similar poly-alphaolefin with a shorter chain length and less branching than the 3004 polymer. We would expect the two substances to exhibit similar toxicological properties.
- 3. The proposed use for this material is still in mechanical filter and mask testing apparatus. In these operations, airborne concentrations of material are low and the potential for human exposure is reduced.
- 4. Based on the structural similarities between Emery 3004 and 3002 and the low potential for human exposures, recommend approval of Emery 3002 for use in "cold smoke" spraying systems.
- 5. POC for this action is Dr. Glenn Leach, (410) 671-3980.

FOR THE COMMANDER:

MAURICE_H. WEEKS

Chief, Toxicology Division

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Enc1 wd SGPS-PSP-E (SMCCR-CMH/3 Sep 92) (40-5e) 1st End LTC Bratt/DSN 289-0125 SUBJECT: Request for Approval for Use of Emery 3002 Army-wide for "Cold Smoke" Spraying Systems

HQDA (SGPS-PSP), 5109 Leesburg Pike, Falls Church, VA 22041-3258 14 October 1992

FOR Commander, US Army Environmental Hygiene Agency,
ATTN: HSHB-MO-T, Aberdeen Proving Ground, MD 21010-5422

- 1. Request the US Army Environmental Hygiene Agency review the enclosed information from the standpoint of toxicity. Direct coordination is authorized. Prepare a recommended position for this office concerning the toxicity clearance of the subject test material.
- 2. Request your recommended position reach this office by 12 January 1993. Staff point of contact for this office is LTC Gary M. Bratt, DSN 289-0125.

Encl nc

ROBERT G. CLAYPOOL Colonel, Medical Corps

Director, Professional Services

CF: (wo/encl)

CDR, AMC, ATTN: AMCSG

CDR, HSC, ATTN: HSCL-P

CDR, MRDC, ATTN: SGRD-PLC

CDR, AMCCOM, ATTN: AMSMC-SG



DEPARTMENT OF THE ARMY U.S. ARMY CHEMICAL RESEARCH, DEVELOPMENT AND ENGINEERING CENTER ABERDEEN PROVING GROUND, MARYLAND 21010-5423



REPLY TO ATTEMPION OF

SMCCR-CMH (40e)

0 3 SEP 1992

MEMORANDUM THRU Commander, U.S. Army Materiel Command,
ATTN: AMCSG), 5001 Eisenhower Avenue,
Alexandria, VA 22333-0001

FOR HQDA (SGPS-PSP), 5111 Leesburg Pike, Falls Church, VA 22041-3258

SUBJECT: Request for Approval for Use of Emery 3002 Army-wide for "Cold Smoke" Spraying Systems

1. References:

- a. CRDEC-TR-370, Implementation of DOP Replacement with Selected Materials in Mask and Filter Testing Penetrometer Machines: Final Report, Jun 92.
- b. Memorandum, HQDA (SGPS-PSP), subject: Request for Approval of Emery 3004 as an Army-Wide Substitute Material for Dioctyl Phthlate (DOP), 8 Jan 92.
- 2. Request your office approve the Army-wide use of Emery 3002, a polyalpha olefin, in "cold smoke" spraying systems.
- 3. Emery 3004 has been approved by your office as a substitute for Dioctyl Phthalate (DOP) (reference 1.b.). It is a mix of polyalpha olefins, one of which is the 20 carbon chain polyolefin predominantly found in Emery 3002 (reference 1.a.).
- 4. A comparison of the percentage of the constituents in Emery 3002 and Emery 3004 is included on page 15 of the enclosure (reference 1.a.). The Material Safety Data Sheet for Emery 3002 is also found on pages A-18 to A-21 of the enclosure (reference 1.a.). The structure of the predominent molecule in Emery 3002 (a C₂₀ polyalpha olefin) is mostly straight-chained and a simpler compound than Emery 3004.
- 5. Our research with "cold smoke" spraying systems (such as the M14 mask face-fit machine and developmental systems using Laskin nozzles (e.g., the HIFATS)), demonstrates Emery 3002 outperforms Emery 3004. Substitution of Emery 3002 for Emery 3004 would further enhance the operation of these systems.

0 3 SEP 1992

SMCCR-CMH (40e)

SUBJECT: Request for Approval for Use of Emery 3002 Army-wide for "Cold Smoke" Spraying Systems

- 6. Funds for any further toxicology research of Emery 3002 do not exist.
- 7. Should your office require any further information please call our point of contact, Mr. Timothy Williams, Senior Industrial Hygienist, SMCCR-CMH, commercial (410) 671-2318, DSN 584-2318.

FOR THE COMMANDER:

Encl

M ROBERT D. PLAKUS

MAJ, MS

Chief, Health and Veterinary

Services Office

APPENDIX B

MATERIAL SAFETY DATA SHEETS (MSDS)
FOR ETHYLFLO 162 AND EMERY 3002

MATERIAL SAFETY DATA SHEET ETHYL CORPORATION - CHEMICALS GROUP FOR EMERGENCIES ONLY - PHONE 504/344-7147 FOR NON EMERGENCIES -- PHONE 504/388-7717

HMIS CLASSIFICATIONS:

HEALTH: 0

FLAMMABILITY: 0 REACTIVITY: 0

ISSUE DATE: 10/30/90

SUPERSEDES: 08/16/90 REFORMATTED: 09/11/91

14.0.22

PRODUCT IDENTIFICATION

PRODUCT NAME: ETHYLFLO 162 Polyalphaolefins

CHEMICAL NAME: 1-Decene, dimer, hydrogenated

CAS NO.: 68649-11-6

CHEMICAL FORMULA: CnH2n+2

CHEMICAL FAMILY: Paraffin hydrocarbon

ETHYLFLO is a trademark of Ethyl Corporation.

COMPONENTS

CAS NO. NOTE EXPOSURE LIMIT CHEMICAL NAME

1-Decene, dimer, hydrogenated 68649-11-6 ND Not established by OSHA/ACGIH.

NOTE: Carcinogenicity listing of components at concentrations greater than or equal to 0.1% indicated by: @=NTP; #=IARC; &=OSHA; +=ACGIH; *=OTHER; ND=Not Designated.

CHEMICAL AND PHYSICAL PROPERTIES

APPEARANCE/ODOR: Colorless liquid/odorless.

VAPOR PRESSURE: < 1.0 mm Hg @ 20C/68F.

SOLUBILITY IN WATER: Negligible.

SPECIFIC GRAVITY: 0.80 @ 15.6/15.6C.

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 162 Polyalphaolefins

14.0.22

FIRE AND EXPLOSION HAZARDS

FLASH POINT (METHOD): 160C/320F (PMCC).

FLAMMABLE LIMITS: Not established.

EXTINGUISHING MEDIA: Dry chemical, water spray (fog), foam or carbon dioxide.

HAZARDOUS THERMAL DECOMPOSITION PRODUCTS: Include oxides of carbon.

SPECIAL FIRE FIGHTING PROCEDURES: As for petroleum products. Use self-contained breathing apparatus.

UNUSUAL FIRE AND EXPLOSION HAZARDS: None known.

REACTIVITY DATA

STABILITY: Stable.

CONDITIONS TO AVOID: None known.

MATERIALS TO AVOID: None known.

HAZARDOUS POLYMERIZATION: Will not occur.

HEALTH HAZARDS

INHALATION: Inhalation of oil mist or vapors at elevated temperature may cause respiratory irritation.

EYE CONTACT: Not expected to be an eye irritant.

SKIN CONTACT: Not expected to be a skin irritant.

INGESTION: Harmful if aspirated into the lungs-do not induce vomiting.

CHRONIC EFFECTS OF OVEREXPOSURE: None known.

EMERGENCY FIRST AID PROCEDURES

INHALATION: If inhaled, remove to fresh air.

10/30/90

EMERGENCY PHONE NUMBER PRODUCT NAME:

(504) 344-7147

ETHYLFLO 162
Polvalphaolefins

14.0.22

EMERGENCY FIRST AID PROCEDURES (Con't)

EYE CONTACT: Begin immediate eye irrigation with cool water.

SKIN CONTACT: Wash contaminated areas with soap and water.

INGESTION: If swallowed, give two glasses of water. Do not induce vomiting.

EXPOSURE CONTROL INFORMATION

EXPOSURE LIMITS: Not established by OSHA/ACGIH.

EYE PROTECTION: Chemical goggles or safety glasses.

PROTECTIVE GLOVES: Resistant to chemical penetration.

RESPIRATORY PROTECTION: NIOSH approved organic vapor respirator when exposed to vapor from heated material.

LOCAL EXHAUST VENTILATION: At bulk vessel openings when handling heated materials.

MECHANICAL VENTILATION: Recommended.

OTHER: If repeated or prolonged skin contact or contamination of clothing is likely, protective clothing should be worn.

ENVIRONMENTAL PROTECTION

SPILLS OR LEAKS: Contain any spills with dikes or absorbents to prevent migration and entry into sewers or streams. Take up small spills with dry chemical absorbent. Large spills may be taken up with pump or vacuum and finished off with dry chemical absorbent. May require excavation of contaminated soil.

DISPOSAL METHODS: To the best of Ethyl's knowledge, this product is not regulated by CERCLA/RCRA as a hazardous waste or material. However, this product has not been tested for the toxicity characteristic via the Toxicity Characteristic Leaching Procedure. Therefore, it may be

10/30/90

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 162 Polyalphaolefins

14.0.22

ENVIRONMENTAL PROTECTION (Con't)

disposed of as an industrial waste in a manner acceptable to good waste management practice and in compliance with applicable local, state and federal regulations.

STORAGE REQUIREMENT: Short term - (less than 24 hours) 65C maximum. Long term - (greater than 24 hours) 50C Maintain product above 10C for flowability.

_______ REGULATORY INFORMATION

TSCA:

THIS MATERIAL IS IN COMPLIANCE WITH THE TOXIC SUBSTANCES CONTROL ACT (15 USC 2601 - 2629).

DOT DESCRIPTION/PROPER SHIPPING NAME:

Not regulated for transportation.

HAZARD CATEGORIES FOR SARA 311/312 REPORTING ARE INDICATED BELOW:

HEALTH Immediate (Acute) HEALTH Delayed (Chronic) No PHYSICAL Fire No Sudden Release of Pressure PHYSICAL No PHYSICAL Reactive No Nuisance Mist/Dust Only No

FOLLOWING ARE WHMIS CLASSIFICATIONS FOR THIS PRODUCT: NOT CONTROLLED

See ''Explanation of Terminology'' for regulations addressed.

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 162 Polyalphaolefins

14.0.22

MSDS prepared by: Health & Environment Department

Ethyl Corporation

FOR ADDITIONAL NONEMERGENCY HEALTH AND SAFETY INFORMATION, C

HEALTH AND ENVIRONMENT DEPARTMENT ETHYL CORPORATION 451 FLORIDA ST. BATON ROUGE, LA. 70801 (800) 535-3030

FOR PRODUCT INFORMATION CALL

(800) 535-3030

THIS MATERIAL SAFETY DATA SHEET CONTAINS AT LEAST THE INFORMATION REQUIRED BY THE FEDERAL OSHA HAZARD COMMUNICATION RULE, 29 CFR 1910.1200(g) (2).

EXPLANATION OF MATERIAL SAFETY DATA SHEET TERMINOLOGY

TLV: Threshold Limit Value PEL: Permissible Exposure Limit TWA8: Timeweighted average concentration for a normal 8-hour workday Short-Term Exposure Limit--a 15 minute avg exposure not to be exceeded STEL: National Institute for Occupational Safety and Health NIOSH: AIHA WEEL: Amer. Industrial Hygiene Assoc. Worker Environmental Exposure Limi NTP: National Toxicology Program International Agency for Research on Cancer IARC: May include preliminary data or studies not evaluated by other agencie OTHER: ACGIH: American Conference of Governmental Industrial Hygienists Occupational Safety and Health Administration OSHA: APPEARANCE/ODOR: Description of material at normal temperature and pressure. BOILING POINT: Temperature at which the liquid boils. MELTING POINT: Temperature at which a substance changes from solid to liquid. VAPOR PRESSURE: The pressure exerted at any temperature by a vapor. SOLUBILITY IN WATER: The amount of the product that will dissolve in water. SPECIFIC GRAVITY: Ratio of the product to an equal volume of water. **EVAPORATION RATE:** Ratio of the vaporization to a known material. PERCENT VOLATILES: The percent of the product that will evaporate. POUR POINT: The lowest temperature at which a liquid will flow from container VISCOSITY: A measure of flow characteristics of a liquid. ----- FIRE AND EXPLOSION HAZARDS ---FLASH POINT (CLOSED CUP METHOD): Lowest temperature at which ignition occurs. FLAMMABLE LIMITS: Range of vapor concentrations at which the product will burn or explode in the presence of a flame. The lower explosive limit The upper explosive limit LEL: UEL: EXTINGUISHING MEDIA: Recommended fire fighting agents. HAZARDOUS THERMAL DECOMPOSITION PRODUCTS: Known hazardous chemicals resulting from heating. UNUSUAL FIRE AND EXPLOSION HAZARDS: Hazards pertaining to chemical reactions in the presence of heat. REACTIVITY DATA -----STABILITY: Indicates susceptibility of the product to dangerously decompose. CONDITIONS AND MATERIALS TO AVOID: Conditions and materials that may cause undesirable reactions of instability. HAZARDOUS DECOMPOSITION PRODUCTS: The hazardous materials produced from a chemical reaction. HAZARDOUS POLYMERIZATION: Tendency to undergo a reaction releasing excess pressure and heat. INHALATION/EYE/SKIN/INGESTION: Immediate symptoms and effects of overexposure by skin or eye contact, breathing vapors of dust, and ingestion. CHRONIC EFFECTS: Effects that may occur a ter repeated or prolonged or effects that may be long lasting after acute exposure. OTHER HEALTH EFFECTS: Includes medical conditions which may be aggravated by exposure to the product and hazards identified with components of of a mixture. TOXICITY: LD50 or LC50 is the dose level that kills half of the animals teste ----- EMERGENCY FIRST AID ------INHALATION/EYE/SKIN/INGESTION: Emergency & first aid instructions for handlin overexposure by inhalation, ingestion, and skin and eye contact. NOTE TO PHYSICIAN: Gives licensed health care professional information on contraindicated or recommended treatment.

EYE PROTECTION: Necessary eye or face protection.

PRCTECTIVE GLOVES: Indicates the need for and type of protective gloves. RESPIRATORY PROTECTION: Respirator recommended for use during routine or

emergency situations.

Type (local/general) of ventilation recommended to capture **VENTILATION:** contaminants or preven the build-up of hazardous atmospheres

OTHER: Other recommended personal protective equipment.

---- ENVIRONMENTAL PROTECTION ----SPILLS AND LEAKS: Special precautions for clean-up of spills and leaks and preparation of chemical for disposal.

DISPOSAL METHOD: EPA classification and proper disposal procedure.

EPA: Environmental Protection Agency.

RCRA: Resource Conservation and Recover Act

STORAGE REQUIREMENTS: Any unusual requirements or precautions for storage.

----- REGULATORY INFORMATION ------CERCLA: Comprehensive Environmental Response, Compensation and Liability Act.

DOT: Department of Transportation.

HTPO: Higher Threshold Planning Quantity. LTPQ: Lower Threshold Planning Quantity.

NJRTK: Determined by New Jersey to be a hazardous substance when present at concentrations greater than or equal to 1.0%

Determined by New Jersey to pose a special health hazard, when NJRTK-SHH: present at concentrations above 0.1%.

N.O.S.: Not otherwise specified.

PA. RTK (PENNSYLVANIA): Determined by Pennsylvania to be hazardous when present at concentrations greater than or equal to 1.0%.

PA. RTK-SHH (PENNSYLVANIA): Determined by Pennsylvania to be hazardous when present at concentrations greater than .01%.

PA. RTK-E (PENNSYLVANIA): Determined by Pennsylvania to be hazardous to the environment.

PROP 65-CA1 (CALIFORNIA): Determined by California to cause cancer.

PROP 65-CA2 (CALIFORNIA): Determined by Calif. to cause reproductive toxicity RQ: Reportable Quantity.

Superfund Amendment Reauthorization Act. SARA:

SARA 302: An extremely hazardous substance listed in 40 CFR355.

SARA 313: Listed in 40 CFR 372.65. Has a de minimis cutoff of 1.0%.

SARA 313-CA: Listed in 40 CFR 372.65. Has a de minimis cutoff of 0.1%.

Canadian Workplace Hazardous Materials Information System.

WHMIS-CN1 (CANADA): On Canada's ingredient disclosure list (IDL) at 1.0%. WHMIS-CN2 (CANADA): On Canada's ingredient disclosure list (IDL) at 0.1%.

WHMIS HC1: Hazardous under WHMIS at a threshold level of 1%.

WHMIS HC.1: Hazardous under WHMIS at a threshold level of .1%.

Although the information and recommendations set forth herein (hereinafter "Information") are presented in good faith and believed to be correct as of th date hereof, Ethyl Corporation makes no representations as to the completeness or accuracy thereof. Information is supplied upon the condition that the persons receiving same will make their own determination as to its safety and suitability for their purposes prior to use. In no event will Ethyl Corporati be responsible for damages of any nature whatsoever resulting from the use or . reliance upon information. NO REPRESENTATIONS OF WARRANTIES, EITHER EXPRESSED OR IMPLIED, OR MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE OR OF ANY OTHER NATURE, ARE MADE HEREUNDER WITH RESPECT TO INFORMATION OR THE PRODUCT TO WHICH THE INFORMATION REFERS.

EMERY CHEMICALS

DIVISION OF NATIONAL DISTILLERS AND CHEMICAL CORPORATION

Material Safety Data Sheet

EMERGENCY PHONE: (513) 482-2297

CHEMTREC 800-424-9300

MSDS REFERENCE: EMERY 3002 (2/25/87)

SECTION I - IDENTIFICATION

PRODUCT:

LEMERY 3002 SYNTHETIC HYDROCARBON 2 CST FLUID

SYNONYMS:

APOLYAL PHAOLEFIN

CHEMICAL:

SYNTHETIC ALIPHATIC HYDROCARBON

CAS NO:

68649-11-6

SECTION II - HAZARDOUS INGREDIENTS

COMPOSITION

PEL/TLY

HAZ ARD

POLYALPHAOLEFIN (68649-11-6)

% 100

NONE/NONE

NONE NOTED

: SECTION III - HEALTH INFORMATION

INHAL ATION:

UNKNOWN

INGESTION:

UNKNOWN

EYE CONTACT:

A PRODUCT CONTAINING MORE THAN 50% POLYALPHAOLEFINS WAS NOT

CLASSIFIED AS AN IRRITANT BY OCULAR APPLICATION.

SKIN CONTACT:

A PRODUCT CONTAINING MORE THAN 50% POLYALPHAOLEFINS WAS NOT

CLASSIFIED AS A PRIMARY IRRITANT OR AS A CORROSIVE BY DERMAL

APPLICATION.

SECTION IV - OCCUPATIONAL EXPOSURE LIMITS

PEL: NO OSHA PEL

TLY: NO ACGIH TLY

SECTION Y - EMERGENCY FIRST AID PROCEDURE

FOR OVEREXPOSURE BY SWALLOWING: DO NOT INDUCE VOMITING. IF VICTIM IS CONSCIOUS AND ABLE TO SWALLOW, PROMPTLY HAVE VICTIM DRINK WATER TO DILUTE. DO NOT GIVE SODIUM BICARBONATE, FRUIT JUICES OR VINEGAR. NEVER GIVE ANYTHING BY MOUTH IF THE VICTIM IS UNCONSCIOUS OR HAVING CONVULSIONS. CALL A PHYSICIAN OR POISON CONTROL CENTER IMMEDIATELY.

FOR OVEREXPOSURE BY SKIN CONTACT: WASH AFFECTED AREA.

FOR OVEREXPOSURE BY EYE CONTACT: IMMEDIATELY FLUSH EYES WITH PLENTY OF COOL WATER FOR AT LEAST 15 MINUTES. DO NOT LET VICTIM RUB EYES.

FOR OVEREXPOSURE BY INHALATION: IMMEDIATELY REMOVE VICTIM TO FRESH AIR. IF VICTIM HAS STOPPED BREATHING GIVE ARTIFICIAL RESPIRATION, PREFERABLY BY MOUTH- TO-MOUTH. GET MEDICAL ATTENTION IMMEDIATELY.

SECTION VI - PHYSICAL DATA

BOILING POINT: INITIAL BOILING POINT 310 DEG C (ASTM D-1078)

VAPOR PRESSURE: <1 MM HG PRESSURE AT 20 DEG C SPECIFIC GRAVITY: 0.790 AT 15.6/15.6 DEG C

SOLUBILITY IN WATER: INSOLUBLE

APPEARANCE AND COLOR:

COLORLESS, ODOR FSS FLUID

SECTION VII - FIRE AND EXPLOSION HAZARDS

FLASH POINT & METHOD USED: 320 DEG F (160 DEG C) ASTM D-92 FLAMMABLE LIMITS IN AIR, \$ BY VOL. LOWER: NOT DETERMINED FLAMMABLE LIMITS IN AIR, \$ BY VOL. UPPER: NOT DETERMINED

NFPA RATING: NO NFPA RATING

SPECIAL FIRE FIGHTING PROCEDURES & PRECAUTIONS

(INDIVIDUALS SHOULD PERFORM ONLY THOSE FIRE FIGHTING PROCEDURES FOR WHICH THEY HAVE BEEN TRAINED). USE WATER SPRAY, DRY CHEMICAL, FOAM OR CARBON DIOXIDE. WATER MAY BE INEFFECTIVE BUT, SHOULD BE USED TO KEEP FIRE-EXPOSED CONTAINERS COOL. IF A SPILL OR LEAK HAS NOT IGNITED, USE WATER SPRAY TO DISPERSE THE VAPORS. WATER SPRAY MAY BE USED TO FLUSH SPILLS AWAY FROM FIRE

UNUSUAL FIRE & EXPLOSION HAZARDS

FIREFIGHTERS SHOULD WEAR SELF-CONTAINED BREATHING APPARATUS IN THE POSITIVE-PRESSURE MODE WITH A FULL FACEPIECE WHEN THERE IS A POSSIBILITY OF EXPOSURE TO SMOKE, FUMES OR HAZARDOUS DECOMPOSITION PRODUCTS.

SECTION VIII - REACTIVITY

STABIL ITY:

GENERALLY STABLE

HAZ ARDOUS POLYMERIZATION:

NONE LIKELY

CONDITIONS & MATERIALS TO AVOID:

AVOID HEATING TO DECOMPOSITION. THE USER IS ADVISED TO HAVE A SAFETY EXPERT EVALUATE THE SPECIFIC CONDITIONS OF USE.

HAZARDOUS DECOMPOSITION PRODUCTS:

DECOMPOSITION OF ORGANIC MATERIALS MAY PRODUCE CARBON MONOXIDE AND CARBON DIOXIDE.

SECTION IX - EMPLOYEE PROTECTION

CONTROL MEASURES:

HANDLE IN THE PRESENCE OF ADEQUATE VENTILATION.

RESPIRATORY PROTECTION:

WHERE EXPOSURE IS LIKELY TO EXCEED ACCEPTABLE CRITERIA (SEE SECTIONS II AND IV), USE NIOSH/OSHA APPROVED RESPIRATORY EQUIPMENT. RESPIRATORS SHOULD BE SELECTED BASED ON THE FORM AND CONCENTRATION OF CONTAMINANT IN AIR AND IN ACCORDANCE WITH OSHA (29 CFR 1910.134).

PROTECTIVE CLOTHING:

WEAR GLOVES AND PROTECTIVE CLOTHING WHICH ARE IMPERVIOUS TO THE PRODUCT FOR THE DURATION OF ANTICIPATED EXPOSURE IF THERE IS POTENTIAL FOR PROLONGED OR REPEATED SKIN CONTACT.

EYE PROTECTION:

WEAR SAFETY GLASSES MEETING THE SPECIFICATIONS OF ANSI STANDARD 287.1.

SECTION X - ENVIRONMENTAL PROTECTION

ENVIRONMENTAL PRECAUTIONS:

AVOID UNCONTROLLED RELEASES OF THIS MATERIAL. WHERE SPILLS ARE POSSIBLE, A COMPREHENSIVE SPILL RESPONSE PLAN SHOULD BE DEVELOPED AND IMPLEMENTED.

SPILL OR LEAK PRECAUTIONS:

WEAR APPROPRIATE RESPIRATORY PROTECTION AND PROTECTIVE CLOTHING AS DESCRIBED IN SECTION IX. CONTAIN SPILLED MATERIAL. TRANSFER TO SECURE CONTAINERS. WHERE NECESSARY, COLLECT USING ABSORBENT MEDIA. IN THE EVENT OF AN UNCONTROLLED RELEASE OF THIS MATERIAL, THE USER SHOULD DETERMINE IF THE RELEASE IS REPORTABLE UNDER APPLICABLE LAWS AND REGULATIONS.

WASTE DISPOSAL:

ALL RECOVERED MATERIAL SHOULD BE PACKAGED, LABELED, TRANSPORTED, AND DISPOSED OR RECLAIMED IN CONFORMANCE WITH APPLICABLE LAWS AND REGULATIONS AND IN CONFORMANCE WITH GOOD ENGINEERING PRACTICES. AVOID LANDFILLING OF LIQUIDS. RECLAIM WHERE POSSIBLE.

SECTION XI - REGULATORY CONTROLS

DEPARTMENT OF TRANSPORTATION:

DOT CLASSIFICATION: NOT REGULATED

DOT PROPER SHIPPING NAME:

OTHER DOT INFORMATION:

OTHER REGULATORY REQUIREMENTS:

LISTED IN TSCA INVENTORY

SECTION XII - PRECAUTIONS: HANDLING, STORAGE AND USAGE NO SPECIAL PRECAUTIONS NECESSARY.

The information presented herein is believed to be factual as it has been derived from the works and opinions of persons believed to be qualified experts; however, nothing contained in this information is to be taken as a warranty or representation for which National Distillers and Chemical Corporation bears legal responsibility. The user should review any recommendations in the specific context of the intended use to determine whether they are appropriate.

PREPARED BY: IRWIN S. SCHLOSSMAN

DATE: 2/25/87 SUPERSEDES: 11/13/86

Emery Chemicals 4900 Este Avenue Cincinnati, Ohio 45232

APPENDIX C

MATERIAL SAFETY DATA SHEETS (MSDS)

FOR ETHYLFLO AND EMERY 3004

MATERIAL SAFETY DATA SHEET ETHYL CORPORATION - CHEMICALS GROUP FOR EMERGENCIES ONLY - PHONE 504/344-7147 FOR NON EMERGENCIES -- PHONE 504/388-7717

HMIS CLASSIFICATIONS:

ISSUE DATE: 10/30/90

HEALTH: 0

SUPERSEDES: 08/16/90 REFORMATTED: 09/11/91

FLAMMABILITY: 0

REACTIVITY: 0

14.0.23

PRODUCT IDENTIFICATION

PRODUCT NAME: ETHYLFLO 164 Polyalphaolefins

CHEMICAL NAME: 1-Decene, homopolymer, hydrogenated

CAS NO.: 68037-01-4

CHEMICAL FORMULA: CnH2n+2

CHEMICAL FAMILY: Paraffin hydrocarbon

ETHYLFLO is a trademark of Ethyl Corporation.

_______ COMPONENTS

CHEMICAL NAME CAS NO. NOTE EXPOSURE LIMIT

1-Decene, homopolymer, 68037-01-4 ND Not established hydrogenated by OSHA/ACGIH.

NOTE: Carcinogenicity listing of components at concentrations greater than or equal to 0.1% indicated by: @=NTP; #=IARC; &=OSHA; +=ACGIH; *=OTHER; ND=Not Designated.

CHEMICAL AND PHYSICAL PROPERTIES

APPEARANCE/ODOR: Colorless, odorless liquid.

BOILING POINT: 375-505C/707-941F.

VAPOR PRESSURE: <1mm Hg @ 20C/68F.

SOLUBILITY IN WATER: Negligible.

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 164 Polyalphaolefins

14.0.23

CHEMICAL AND PHYSICAL PROPERTIES (Con't)

SPECIFIC GRAVITY: 0.82 @ 15.6/15.6C.

FIRE AND EXPLOSION HAZARDS

FLASH POINT (METHOD): 224C/435F (PMCC).

FLAMMABLE LIMITS: Not established.

EXTINGUISHING MEDIA: Dry chemical, water spray (fog), foam or carbon dioxide.

HAZARDOUS THERMAL DECOMPOSITION PRODUCTS: Include oxides of carbon.

SPECIAL FIRE FIGHTING PROCEDURES: As for petroleum products. Use self-contained breathing apparatus.

UNUSUAL FIRE AND EXPLOSION HAZARDS: None known.

REACTIVITY DATA

STABILITY: Stable.

HAZARDOUS POLYMERIZATION: Will not occur.

HEALTH HAZARDS

INHALATION: Inhalation of oil mist or vapors at elevated temperature may cause respiratory irritation.

EYE CONTACT: Not expected to be an eye irritant.

SKIN CONTACT: Not expected to be a skin irritant.

INGESTION: Harmful if aspirated into the lungs-do not induce vomiting.

CHRONIC EFFECTS OF OVEREXPOSURE: None known.

EMERGENCY PHONE NUMBER PRODUCT NAME:

(504) 344-7147

ETHYLFLO 164
Polyalphaolefins

14.0.23

EMERGENCY FIRST AID PROCEDURES

INHALATION: If inhaled, remove to fresh air.

EYE CONTACT: Begin immediate eye irrigation with cool water.

SKIN CONTACT: Wash contaminated areas with soap and water.

INGESTION: If swallowed, give two glasses of water. Do not induce vomiting.

EXPOSURE CONTROL INFORMATION

EXPOSURE LIMITS: Not established by OSHA/ACGIH.

EYE PROTECTION: Chemical goggles or safety glasses.

PROTECTIVE GLOVES: Resistant to chemical penetration.

RESPIRATORY PROTECTION: NIOSH approved organic vapor respirator when exposed to vapor from heated material.

LOCAL EXHAUST VENTILATION: At bulk vessel openings when handling heated materials.

MECHANICAL VENTILATION: Recommended.

OTHER: If repeated or prolonged skin contact or contamination of clothing is likely, protective clothing should be worn.

ENVIRONMENTAL PROTECTION

SPILLS OR LEAKS: Contain any spills with dikes or absorbents to prevent migration and entry into sewers or streams.

Take up small spills with dry chemical absorbent.

Large spills may be taken up with pump or vacuum and finished off with dry chemical absorbent. May require excavation of contaminated soil.

DISPOSAL METHODS: To the best of Ethyl's knowledge, this product is not regulated by CERCLA/RCRA as a hazardous waste or material. However, this product has not been

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 164 Polyalphaolefins

ENVIRONMENTAL PROTECTION (Con't)

tested for the toxicity characteristic via the Toxicity Characteristic Leaching Procedure. Therefore, it may be disposed of as an industrial waste in a manner acceptable to good waste management practice and in compliance with applicable local, state and federal regulations.

STORAGE REQUIREMENT: Short term - (less than 24 hours) 65C maximum. Long term - (greater than 24 hours) 50C Maintain product above 10C for flowability. maximum.

REGULATORY INFORMATION

TSCA:

THIS MATERIAL IS IN COMPLIANCE WITH THE TOXIC SUBSTANCES CONTROL ACT (15 USC 2601 - 2629).

DOT DESCRIPTION/PROPER SHIPPING NAME:

Not regulated for transportation.

HAZARD CATEGORIES FOR SARA 311/312 REPORTING ARE INDICATED BELOW:

HEALTH Immediate (Acute) No HEALTH Delayed (Chronic) No PHYSICAL Fire No PHYSICAL Sudden Release of Pressure No PHYSICAL Reactive No Nuisance Mist/Dust Only No

FOLLOWING ARE WHMIS CLASSIFICATIONS FOR THIS PRODUCT: NOT CONTROLLED

See 'Explanation of Terminology' for regulations addressed.

EMERGENCY PHONE NUMBER PRODUCT NAME: (504) 344-7147

ETHYLFLO 164 Polyalphaolefins

14.0.23

MSDS prepared by: Health & Environment Department Ethyl Corporation

FOR ADDITIONAL NONEMERGENCY HEALTH AND SAFETY INFORMATION, C

HEALTH AND ENVIRONMENT DEPARTMENT ETHYL CORPORATION 451 FLORIDA ST. BATON ROUGE, LA. 70801 (800) 535-3030

FOR PRODUCT INFORMATION CALL (800) 535-3030

THIS MATERIAL SAFETY DATA SHEET CONTAINS AT LEAST THE INFORMATION REQUIRED BY THE FEDERAL OSHA HAZARD COMMUNICATION RULE, 29 CFR 1910.1200(g) (2).

EXPLANATION OF MATERIAL SAFETY DATA SHEET TERMINOLOGY

```
----- HAZARDOUS COMPONENTS ---------
TLV: Threshold Limit Value
PEL:
       Permissible Exposure Limit
TWA8: Timeweighted average concentration for a normal 8-hour workday
        Short-Term Exposure Limit--a 15 minute avg exposure not to be exceeded
STEL:
NIOSH: National Institute for Occupational Safety and Health
AIHA WEEL: Amer. Industrial Hygiene Assoc. Worker Environmental Exposure Limi
       National Toxicology Program
       International Agency for Research on Cancer
OTHER: May include preliminary data or studies not evaluated by other agencie
ACGIH: American Conference of Governmental Industrial Hygienists
OSHA: Occupational Safety and Health Administration
APPEARANCE/ODOR: Description of material at normal temperature and pressure.
BOILING POINT: Temperature at which the liquid boils.
MELTING POINT: Temperature at which a substance changes from solid to liquid. VAPOR PRESSURE: The pressure exerted at any temperature by a vapor.
SOLUBILITY IN WATER: The amount of the product that will dissolve in water.
SPECIFIC GRAVITY: Ratio of the product to an equal volume of water.

EVAPORATION RATE: Ratio of the vaporization to a known material.

PERCENT VOLATILES: The percent of the product that will evaporate.

POUR POINT: The lowest temperature at which a liquid will flow from container VISCOSITY: A measure of flow characteristics of a liquid.
 ------FIRE AND EXPLOSION HAZARDS ---------
FLASH POINT (CLOSED CUP METHOD): Lowest temperature at which ignition occurs. FLAMMABLE LIMITS: Range of vapor concentrations at which the product will
        burn or explode in the presence of a flame.
        The lower explosive limit
UEL:
        The upper explosive limit
EXTINGUISHING MEDIA: Recommended fire fighting agents.
HAZARDOUS THERMAL DECOMPOSITION PRODUCTS: Known hazardous chemicals resulting
        from heating.
UNUSUAL FIRE AND EXPLOSION HAZARDS: Hazards pertaining to chemical reactions
        in the presence of heat.
                                   REACTIVITY DATA -----
      -----
STABILITY: Indicates susceptibility of the product to dangerously decompose. CONDITIONS AND MATERIALS TO AVOID: Conditions and materials that may cause
        undesirable reactions of instability.
HAZARDOUS DECOMPOSITION PRODUCTS: The hazardous materials produced from a
        chemical reaction.
HAZARDOUS POLYMERIZATION: Tendency to undergo a reaction releasing excess
     pressure and heat.
----- HEALTH HAZARDS ------
INHALATION/EYE/SKIN/INGESTION: Immediate symptoms and effects of overexposure by skin or eye contact, breathing vapors of dust, and ingestion.
CHRONIC EFFECTS: Effects that may occur after repeated or prolonged
        or effects that may be long lasting after acute exposure.
OTHER HEALTH EFFECTS: Includes medical conditions which may be aggravated by
        exposure to the product and hazards identified with components of
        of a mixture.
TOXICITY: LD50 or LC50 is the dose level that kills half of the animals teste
INHALATION/EYE/SKIN/INGESTION: Emergency & first aid instructions for handlin
        overexposure by inhalation, ingestion, and skin and eye contact.
NOTE TO PHYSICIAN: Gives licensed health care professional information on
        contraindicated or recommended treatment.
      ----- EXPOSURE CONTROL INFORMATION ---------
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EYE PROTECTION: Necessary eye or face protection.

PROTECTIVE GLOVES: Indicates the need for and type of protective gloves. RESPIRATORY PROTECTION: Respirator recommended for use during routine or emergency situations.

VENTILATION: Type (local/general) of ventilation recommended to capture contaminants or preven the build-up of hazardous atmospheres

OTHER: Other recommended personal protective equipment.

----- ENVIRONMENTAL PROTECTION ----

SPILLS AND LEAKS: Special precautions for clean-up of spills and leaks and preparation of chemical for disposal.

DISPOSAL METHOD: EPA classification and proper disposal procedure.

EPA: Environmental Protection Agency.

RCRA: Resource Conservation and Recover Act

STORAGE REQUIREMENTS: Any unusual requirements or precautions for storage. ----- REGULATORY INFORMATION -----

CERCLA: Comprehensive Environmental Response, Compensation and Liability Act.

DOT: Department of Transportation.

Higher Threshold Planning Quantity. HTPO: LTPO: Lower Threshold Planning Quantity.

NJRTK: Determined by New Jersey to be a hazardous substance when present at concentrations greater than or equal to 1.0%

NJRTK-SHH: Determined by New Jersey to pose a special health hazard, when present at concentrations above 0.1%.

N.O.S.: Not otherwise specified.

PA. RTK (PENNSYLVANIA): Determined by Pennsylvania to be hazardous when present at concentrations greater than or equal to 1.0%.

PA. RTK-SHH (PENNSYLVANIA): Determined by Pennsylvania to be hazardous when present at concentrations greater than .01%.

PA. RTK-E (PENNSYLVANIA): Determined by Pennsylvania to be hazardous to the environment.

PROP 65-CA1 (CALIFORNIA): Determined by California to cause cancer. PROP 65-CA2 (CALIFORNIA): Determined by Calif. to cause reproductive toxicity RQ: Reportable Quantity.

SARA: Superfund Amendment Reauthorization Act.

SARA 302: An extremely hazardous substance listed in 40 CFR355. SARA 313: Listed in 40 CFR 372.65. Has a de minimis cutoff of 1.0%. SARA 313-CA: Listed in 40 CFR 372.65. Has a de minimis cutoff of 0.1%.

WHMIS: Canadian Workplace Hazardous Materials Information System.

WHMIS-CN1 (CANADA): On Canada's ingredient disclosure list (IDL) at 1.0%. WHMIS-CN2 (CANADA): On Canada's ingredient disclosure list (IDL) at 0.1%.

WHMIS HC1: Hazardous under WHMIS at a threshold level of 1%.

WHMIS HC.1: Hazardous under WHMIS at a threshold level of .1%.

Although the information and recommendations set forth herein (hereinafter "Information") are presented in good faith and believed to be correct as of th date hereof, Ethyl Corporation makes no representations as to the completeness or accuracy thereof. Information is supplied upon the condition that the persons receiving same will make their own determination as to its safety and suitability for their purposes prior to use. In no event will Ethyl Corporati be responsible for damages of any nature whatsoever resulting from the use or reliance upon information. NO REPRESENTATIONS OF WARRANTIES, EITHER EXPRESSED OR IMPLIED, OR MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE OR OF ANY OTHER NATURE, ARE MADE HEREUNDER WITH RESPECT TO INFORMATION OR THE PRODUCT TO WHICH THE INFORMATION REFERS.

HENKEL CORPORATION

EMERY GROUP

Material Safety Data Sheet

EMERGENCY PHONE:

(513) 482-2297

CHEMTREC 800-424-9300

MSDS REFERENCE: EMERY 3004 (3/31/89)

SECTION I - IDENTIFICATION

PRODUCT:

EMERY 3004 SYNTHETIC HYDROCARBON 4 CST FLUID

SYNONYMS:

POLYALPHAOLEFIN

CHEMICAL:

SYNTHETIC ALIPHATIC HYDROCARBON

CAS NO:

68649-12-7

SARA HAZARD: NONE NOTED (SECTION 311/312)

TITLE III SECTION 313- NOT LISTED

SECTION II - INGREDIENTS AND HAZARD CLASSIFICATION

COMPOSITION

POLYALPHAOLEFIN (68649-12-7)

100

PEL/TLV NONE/NONE HAZARD

NONE NOTED

SECTION III - HEALTH INFORMATION

INHALATION:

THE ESTIMATED LC50 FOR A 1 HOUR EXPOSURE TO 2 CST PAO WAS 4.68 MG/L (RATS), WHICH IS CONSIDERED TOXIC. IN ORDER TO DETERMINE THE LC50 VALUE, EXTREMELY HEAVY MISTS OF PAO WERE REQUIRED. THE VERY HEAVY MISTS AT THE NECESSARY CONCENTRATION MADE VISIBILITY DIFFICULT AND WOULD BE DIFFICULT TO WORK IN FOR ANY PERIOD OF TIME. THE AUTHOR REPORTED THAT HISTOPATHOLOGICAL CHANGES MAY HAVE BEEN A RESPONSE TO A PHYSICAL INSULT RATHER THAN A SPECIFIC COMPOUND RELATED TOXICITY EFFECT AND THAT 2 CST PAO MAY BE CONSIDERED NON-HAZARDOUS FOR ALL PRACTICAL PURPOSES BY INHALATION.

INGESTION:

THE ACUTE ORAL LD50 VALUE WAS FOUND TO BE GREATER THAN 5.0 G/KG IN MALE AND FEMALE SPRAGUE-DAWLEY RATS. THE MATERIAL IS NOT CLASSIFIED AS TOXIC BY ORAL ADMINISTRATION AS DEFINED IN 16 CFR 1500.

EYE CONTACT:

THE EYES OF 2 RABBITS WERE FOUND TO SHOW EVIDENCE OF CONJUNCTIVAL CHANGES. IRRITATION SCORES IN INDIVIDUAL RABBITS RANGED FROM 0-2 (SCALE 0-110). THE MATERIAL IS NOT CLASSIFIED AS AN IRRITANT BY OCULAR APPLICATION AS DEFINED BY 16 CFR 1500.

SKIN CONTACT:

THE PRIMARY IRRITATION INDEX WAS FOUND TO BE 1.3 (SCALE 0-8) BASED ON ERYTHEMA AND EDEMA. NO EVIDENCE OF TISSUE DAMAGE WAS FOUND. THE MATERIAL IS NOT CLASSIFIED AS A PRIMARY IRRITANT OR AS A CORROSIVE BY DERMAL APPLICATION AS DEFINED BY 16 CFR 1500.

EMERY MSDS

TESTING OF EMERY 3004 FOR COMEDOGENIC POTENTIAL (BLACKHEAD FORMATION) INDICATED THAT UNDER THE CONDITIONS OF THE STUDY, THE MATERIAL WAS NOT CONSIDERED TO SHOW COMEDOGENIC POTENTIAL.

SECTION IV - OCCUPATIONAL EXPOSURE LIMITS

PEL: TWA: 5 MG/M3 (OIL MIST)

TLV: TWA: 5 MG/M3; STEL: 10 MG/M3 (OIL MIST)

SECTION V - EMERGENCY FIRST AID PROCEDURE

FOR OVEREXPOSURE BY <u>SWALLOWING</u>: DO <u>NOT</u> INDUCE VOMITING. IF VICTIM IS CONSCIOUS AND ABLE TO SWALLOW, <u>PROMPTLY</u> HAVE VICTIM DRINK WATER TO DILUTE. DO <u>NOT</u> GIVE SODIUM BICARBONATE, FRUIT JUICES OR VINEGAR. <u>NEVER</u> GIVE ANYTHING BY <u>MOUTH</u> IF THE VICTIM IS UNCONSCIOUS OR HAVING CONVULSIONS. <u>CALL</u> A PHYSICIAN OR POISON CONTROL CENTER IMMEDIATELY.

FOR OVEREXPOSURE BY SKIN CONTACT: WASH AFFECTED AREA.

FOR OVEREXPOSURE BY EYE CONTACT: IMMEDIATELY FLUSH EYES WITH PLENTY OF COOL WATER FOR AT LEAST 15 MINUTES. DO NOT LET VICTIM RUB EYES.

FOR OVEREXPOSURE BY INHALATION: IMMEDIATELY REMOVE VICTIM TO FRESH AIR. IF VICTIM HAS STOPPED BREATHING GIVE ARTIFICIAL RESPIRATION, PREFERABLY BY MOUTH-TO-MOUTH. GET MEDICAL ATTENTION IMMEDIATELY.

SECTION VI - PHYSICAL DATA

BOILING POINT: 754 DEG F (401 DEG C) ASTM D-86 VAPOR PRESSURE: <1 MM HG PRESSURE AT 20 DEG C SPECIFIC GRAVITY: 0.819 AT 15.6/15.6 DEG C SOLUBILITY IN WATER: INSOLUBLE

APPEARANCE AND COLOR:

COLORLESS, ODORLESS FLUID

SECTION VII - FIRE AND EXPLOSION HAZARDS

FLASH POINT & METHOD USED: 432 DEG F (222 DEG C) ASTM D-92 FLAMMABLE LIMITS IN AIR, & BY VOL. LOWER: NOT DETERMINED FLAMMABLE LIMITS IN AIR, & BY VOL. UPPER: NOT DETERMINED

NFPA RATING: NO NFPA RATING

HMIS RATING: HEALTH (0) FIRE (1) REACTIVITY (0)

SPECIAL FIRE FIGHTING PROCEDURES & PRECAUTIONS

(INDIVIDUALS SHOULD PERFORM ONLY THOSE FIRE FIGHTING PROCEDURES FOR WHICH THEY HAVE BEEN TRAINED). USE WATER SPRAY, DRY CHEMICAL, FOAM OR CARBON DIOXIDE. WATER MAY BE INEFFECTIVE BUT SHOULD BE USED TO KEEP FIRE-EXPOSED CONTAINERS COOL. IF A SPILL OR LEAK HAS NOT IGNITED, USE WATER SPRAY TO DISPERSE THE VAPORS. WATER SPRAY MAY BE USED TO FLUSH SPILLS AWAY FROM FIRE.

UNUSUAL FIRE & EXPLOSION HAZARDS

FIREFIGHTERS SHOULD WEAR SELF-CONTAINED BREATHING APPARATUS IN THE POSITIVE-PRESSURE MODE WITH A FULL FACEPIECE WHEN THERE IS A POSSIBILITY OF EXPOSURE TO SMOKE, FUMES OR HAZARDOUS DECOMPOSITION PRODUCTS.

SECTION VIII - REACTIVITY

STABILITY:

GENERALLY STABLE

HAZARDOUS POLYMERIZATION:

NONE LIKELY

CONDITIONS & MATERIALS TO AVOID:

AVOID HEATING TO DECOMPOSITION. THE USER IS ADVISED TO HAVE A SAFETY EXPERT EVALUATE THE SPECIFIC CONDITIONS OF USE.

HAZARDOUS DECOMPOSITION PRODUCTS:

DECOMPOSITION MAY PRODUCE CARBON MONOXIDE AND CARBON DIOXIDE.

SECTION IX - EMPLOYEE PROTECTION

CONTROL MEASURES:

HANDLE IN THE PRESENCE OF ADEQUATE VENTILATION.

RESPIRATORY PROTECTION:

WHERE EXPOSURE IS LIKELY TO EXCEED ACCEPTABLE CRITERIA (SEE SECTIONS II AND IV), USE NIOSH/OSHA APPROVED RESPIRATORY EQUIPMENT. RESPIRATORS SHOULD BE SELECTED BASED ON THE FORM AND CONCENTRATION OF CONTAMINANT IN AIR AND IN ACCORDANCE WITH OSHA (29 CFR 1910.134).

PROTECTIVE CLOTHING:

WEAR GLOVES AND PROTECTIVE CLOTHING WHICH ARE IMPERVIOUS TO THE PRODUCT FOR THE DURATION OF ANTICIPATED EXPOSURE IF THERE IS POTENTIAL FOR PROLONGED OR REPEATED SKIN CONTACT.

EYE PROTECTION:

WEAR SAFETY GLASSES MEETING THE SPECIFICATIONS OF ANSI STANDARD Z87.1.

SECTION X - ENVIRONMENTAL PROTECTION

ENVIRONMENTAL PRECAUTIONS:

AVOID UNCONTROLLED RELEASES OF THIS MATERIAL. WHERE SPILLS ARE POSSIBLE, A COMPREHENSIVE SPILL RESPONSE PLAN SHOULD BE DEVELOPED AND IMPLEMENTED.

SPILL OR LEAK PRECAUTIONS:

WEAR APPROPRIATE RESPIRATORY PROTECTION AND PROTECTIVE CLOTHING AS DESCRIBED IN SECTION IX. CONTAIN SPILLED MATERIAL. TRANSFER TO SECURE CONTAINERS. WHERE NECESSARY, COLLECT USING ABSORBENT MEDIA. IN THE EVENT OF AN UNCONTROLLED RELEASE OF THIS MATERIAL, THE USER SHOULD DETERMINE IF THE RELEASE IS REPORTABLE UNDER APPLICABLE LAWS AND REGULATIONS.

WASTE DISPOSAL:

ALL RECOVERED MATERIAL SHOULD BE PACKAGED, LABELED, TRANSPORTED, AND DISPOSED OR RECLAIMED IN CONFORMANCE WITH APPLICABLE LAWS AND REGULATIONS AND IN CONFORMANCE WITH GOOD ENGINEERING PRACTICES. AVOID LANDFILLING OF LIQUIDS. RECLAIM WHERE POSSIBLE.

SECTION XI - REGULATORY CONTROLS

DEPARTMENT OF TRANSPORTATION:

DOT CLASSIFICATION: NOT REGULATED DOT PROPER SHIPPING NAME: OTHER DOT INFORMATION:

OTHER REGULATORY REQUIREMENTS:

LISTED IN TSCA INVENTORY

SECTION XII - PRECAUTIONS: HANDLING, STORAGE AND USAGE

NO SPECIAL PRECAUTIONS NECESSARY.

The information presented herein is believed to be factual as it has been derived from the works and opinions of persons believed to be qualified experts; however, nothing contained in this information is to be taken as a warranty or representation for which Henkel Corporation bears legal responsibility. The user should review any recommendations in the specific context of the intended use to determine whether they are appropriate.

PREPARED BY: ROBERT E. BORGERDING

DATE: 3/31/89

SUPERSEDES: 11/2/88

Henkel Corporation - Emery Group 4900 Este Avenue Cincinnati, Ohio 45232

APPENDIX D

MANUFACTURER'S SPECIFICATION SHEETS

(ETHYL CORPORATION) FOR POLY-ALPHA OLEFINS (PAOS)



May 24, 1993

Mr. Hugh Carlon U.S. Army, EREDC Bldg. E-3330 Edgewood Area Aberdeen Proving Ground, MD 21010-5423

Dear Mr. Carlon:

In response to your inquiry about Ethyl Corporation's "ETHYLFLO" polyalphaolefins, we have enclosed product literature that highlights the appropriate applications for your requirements.

To enable us to better serve you, Mr. Carlon, we would appreciate your input on the attached survey form. We look forward to hearing from you again and if we can be of assistance, please call us at (800) 535-3030.

Very truly yours,

Denise A. Rizan

Telemarketing Representative

\dar

Enclosures

ETHYLFLO™ 162

Description

ETHYLFLO 162 polyalphaolefin is a hydrogenated synthetic hydrocarbon base fluid for use in fully and partially synthetic, premium, long-drain lubricating oils, industrial oils, hydraulic fluids, transmission fluids or heat transfer fluids.

Features

Thermal stability
Oxidation resistance
Low temperature fluidity

Physical Properties

	TYPICAL	RANGE	TEST METHOD
Appearance	Clear/Bright	İ	Observation
Color	<0.5		ASTM D 1500
Viscosity, cSt, 200°C	0.73		ASTM D 445
Viscosity, cSt, 150°C	1.05		ASTM D 445
Viscosity, cSt, 125°C	1.35		ASTM D 445
Viscosity, cSt, 100°C	1.80	1.55-2.10	ASTM D 445
Viscosity, cSt, 40°C	5.54		ASTM D 445
Viscosity, cSt, -18°C	62.0		ASTM D 445
Viscosity, cSt, -40°C	310		ASTM D 445
Pour Point, °C	< -65	-55 max.	ASTM D 97
Flash Point, °C	>155	145 min.	ASTM D 92
NOACK Volatility			
250°C, 1 hr, % wt. Evap.	99		DIN 51581
Specific Gravity, 15.6/15.6°C (60/60°F)	0.797		ASTM D 1298
Density, lb/gal, 15°C (60°F)	6.653		
Total Acid Number, mg KOH/g		0.01	ASTM D 974
Bromine Number, g Br/100g	0.5	1.0	IP-129
Moisture, ppm	50	75 max.	ASTM D 1744
Molecular Weight	287		GC (calculated)
Dielectric Constant			
23°C, 1.0 and 1000 KHz	2.076		
Decene (C ₁₀) Monomer, % wt.		1.0 max.	GC

ETHYLFLO™ 164

Description

ETHYLFLO 164 polyalphaolefin is a hydrogenated synthetic hydrocarbon base fluid for use in fully and partially synthetic, premium, long-drain lubricating oils, industrial oils, hydraulic fluids, transmission fluids or heat transfer fluids.

Features

Thermal stability
Oxidation resistance
Low volatility

Typical Properties

	TYPICAL	RANGE	TEST METHOD
Appearance	Clear/Bright		Observation
Color	<0.5		ASTM D 1500
Viscosity, cSt, 200°C	1.31		ASTM D 445
Viscosity, cSt, 150°C	1.90		ASTM D 445
Viscosity, cSt, 125°C	2.61		ASTM D 445
Viscosity, cSt, 100°C	3.90	3.8-4.1	ASTM D 445
Viscosity, cSt, 40°C	16.8		ASTM D 445
Viscosity, cSt, -18°C	341		ASTM D 445
Viscosity, cSt, -40°C	2460	2600 max.	ASTM D 445
Viscosity Index	137		ASTM D 2270
Brookfield Viscosity, cP, -40°C	2100		
Cold Cranking Simulator, cP, -25°C	490		
Pour Point, °C	-70	-65 min.	ASTM D 97
Flash Point, °C	215	204 min.	ASTM D 92
Fire Point, °C	250		ASTM D 92
Specific Gravity			
15.6/15.6°C (60/60°F)	0.818		ASTM D 1298
Density, lb/gal, 15.6°C (60°F)	6.81		
Total Acid Number, mg KOH/g	<0.01		ASTM D 974
Bromine Number, g Br/100g	0.2	0.4 max.	IP-129
Moisture, ppm	11	25 max.	ASTM D 1744
Molecular Weight	437		GC (calculated)
NOACK Volatility,			
250°C, 1 hr, % wt. Evap.	12	14 max.	DIN 51581
DSC, Oxidation			
Onset Temperature, °C	192		
DSC, Energy kJ/g	12.1		ASTM D 611
Aniline Point, °C (°F)	116.7 (242)		ASTM D 972
Evaporative Weight Loss, %	11.8		
Dielectric Constant			
23°C, 1.0 and 1000 KHz	2.102		



LUBRICATING PROPERTIES

Four-Ball Wear Test (ASTM D4172)

Average Wear, mm

ETHYLFLO 162 Polyalphaolefin	>2.000
ETHYLFLO 164 Polyalphaolefin	0.719
ETHYLFLO 166 Polyalphaolefin	0.668
ETHYLFLO 168 Polyalphaolefin	0.611
ETHYLFLO 170 Polyalphaolefin	0.549

Conditions: Load = 40 Kg, 75°C, Running Time = 60 minutes, RPM = 600



TYPICAL COMPOSITION

	ETHYLFLO™ Polyalphaolefin				
Composition (norm. area %-GC)	<u>164</u>	<u>166</u>	168	170	
dimer	0.6	0.1		0.1	
-	84.4	33.9	6.0	1.1	
trimer	14.5 •	43.5	55.7	42.5	
tetramer	0.5	17.4	27.2	32.3	
pentamer		3.8	7.0	11.8	
hexamer heptamer		1.3	4.1	12.2	



SIMULATED DISTILLATION

	ETHYLFLO™ Polyalphaolefin				
% Distilled @ °C	<u>164</u>	<u> 166</u>	<u>168</u>	<u>170</u>	
1	369	369	344	424	
5	391	391	402	461	
10	402	402	432	473	
20	412	412	468	479	
50	422	476	491	492	
90	468	525	537	537	
95	476	525	548	542	
99	518	560	560	547	

ASTM D 2887



SEAL COMPATIBILITY

CCMC G5 Specification				
Elastomer: Fluoroelastomer RE1	Tensile Strength % Change	Elongation % Change	Volume % Change	Hardness Change, Pts.
ETHYLFLO 164 Polyalphaolefin	-4.5	-19.0	+1.0	-1.0
ETHYLFLO 166 Polyalphaolefin	-1.2	-13.0	+0.6	0.0
ETHYLFLO 168 Polyalphaolefin	-1.9	-19.0	+0.3	0.0
ETHYLFLO 170 Polyalphaolefin	+1.8	-16.0	+0.1	0.0
Finished Lubricant Limits	-50/0	-60/0	0/+5	0/+5
Elastomer: Acrylate RE2	Tensile Strength % Change	Elongation <u>% Change</u>	Volume <u>% Change</u>	Hardness Change, Pts.
Elastomer: Acrylate RE2 ETHYLFLO 164 Polyalphaolefin	Strength	•		
	Strength % Change	% Change	% Change	Change, Pts.
ETHYLFLO 164 Polyalphaolefin	Strength % Change	% Change -33.0	% Change -0.70	Change, Pts. +3.0
ETHYLFLO 164 Polyalphaolefin ETHYLFLO 166 Polyalphaolefin	Strength % Change -16.0 -12.0	% Change -33.0 -30.0	% Change -0.70 -1.50	Change, Pts. +3.0 +4.0



GLOSSARY OF TERMS AND METHODS

Aniline point (ASTM D 611): It is most often used to provide an estimate of the aromatic hydrocarbon content of mixtures. The aniline point of a sample is the minimum equilibrium solution temperature for equal volumes of aniline and the sample. For example, aromatic hydrocarbons exhibit the lowest aniline point values and paraffins the highest value.

Bromine Number (ASTM D 1158): Bromine number, a measure of aliphatic unsaturation, is the number of grams of bromine that will react with 100 grams of the sample under test conditions.

<u>CCMC G5 Specification*</u>: The limits for this test define the minimum quality level of a finished lubricant for presentation to CCMC Members. The method specifies the immersion of the seal material in the test fluid for seven days at 150°C without preaging (CEC L-39-T-87).

Cold Cranking Simulator (ASTM D 2602): Viscosities measured at -18°C in a special rotary viscometer, the Cold Cranking Simulator (CCS), serve as the basis for the SAE classification of oils.

<u>Dielectric Constant*</u>: The dielectric constant is used to measure fluid polarity. For example, the dielectric constant of hexane is 1.9 whereas the dielectric constant of water is 78. The values for ETHYLFLO PAO were measured at 1.0KHZ and at 1000 KHZ. The results for each liquid at the two frequencies were very close together, and an average value is reported.

<u>Differential Scanning Calorimetry (DSC)*</u>: DSC is used to determine the onset oxidation temperature of the sample fluid using a Du Pont 912 DSC. The sample is heated at a rate of 10°C/minute from 25°C to 500°C in a sealed aluminum pan containing 500 psi of oxygen.

<u>Fire Point (ASTM D 92)</u>: Fire point is defined as the lowest temperature at which a sample will sustain burning for five seconds. It is a measure of the tendency of the test material to form a flammable mixture with air under controlled laboratory conditions.

Flash Point (ASTM D 92): Flash point measures the tendency of the sample to form a flammable mixture with air under controlled laboratory conditions. It is the lowest temperature corrected to 760 mm Hg, at which the application of a test flame causes the vapor of the fluid to ignite.

<u>Four-Ball Wear Test (ASTM D 4172)</u>: This test is used to determine the relative wear preventive properties of lubricating fluids in sliding contact under the prescribed test conditions.

Blank

APPENDIX E

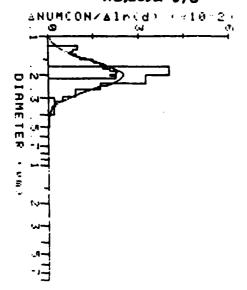
TYPICAL PERFORMANCE DATA:
HOT SMOKE APPLICATIONS

EMERY 3004

49,800 A 10,2 0 00 0	
DIAMEIFF CUM.	6 170 2940 1 81E+002 1 200 3414 2 44E+002 2 230 2171 1 77E+002 3 260 1013 9 28E+001 4 290 404 4 10E+001 5 320 135 1 51E+001 6 350 43 5 23E+000 7 380 9 1 18E+000 8 410 1 1 42E-001 9 440 0 0 00E+000 10 470 0 0 00E+000 11 500 0 0 00E+000 12 530 0 0 00E+000 13 560 0 0 00E+000 14 590 0 00E+000 15 500 0 0 00E+000 16 500 1 = OVERCOUNT)
DATE 90/3/30 TIME 132600 OTHOMAX CNTS/SEC= 598 SEC= 20 R:0-3 TPVN 0 BINS 5 00 ML SEC DILUTION FATIO = , 100E+000 TEMPERATURE (C)= 2.100E+001 TEMPERATURE (mm He)= 7.500E+001 REL HUMIDITY (%)= 5.600E+001 RPRAMETER = 0.000E+000 CARAMETER = 0.000E+000 CARA	PROBE RANGE=1 TOT CNTS= 52: BIN DIA COUNT DISTN VALUE 0 300 516 1.79E+001 1 400 4 1.79E-001 2 500 0 0.00E+000 3 600 1 6.49E-002 4 700 0 0.00E+000 5 800 0 0.00E+000 7 1.000 0 0.00E+000 8 1.100 0 0.00E+000 9 1.200 0 0.00E+000 10 1.300 0 0.00E+000 11 1.400 0 0.00E+000 12 1.500 0 0.00E+000 13 1.600 0 0.00E+000 14 1.700 0 0.00E+000 1 9=OVERCOUNT)
FIT NUM CONC (#/cm2) = 0 0002-000 FIT GEOM MN DIA (##) = 1 0002-000 FIT GEOM STAND DEV = 1 0002-000 PROBE RANGE = 7 TOT CNTS = 10002-000 PROBE RANGE = 7 TOT CNTS = 10002-000 120 382 7.83E+001 1 126 273 5.01E+001 1 126 273 5.01E+001 3 138 171 7.08E+001 4 144 128 7.14E+001 4 144 128 7.14E+001 5 150 160 7.000 6 156 229 6077 2.000 7 162 303 9 73E+001 10 180 506 1 94E+001 10 180 506 1 94E+001 11 186 611 1 92E+002 12 192 677 2 19E+002 13 198 659 2 74E+002 14 204 817 2 82E+001 14 204 817 2 82E+001	PROBE RANGE=0 TOT CHTS= 0 BIN OIR COUNT DISTN VALUE 0 1500 0 00E+000 1 1900 0 00E+000 2 2 300 0 0 00E+000 3 2 700 0 0 00E+000 4 3 100 0 0 00E+000 5 3 500 0 0 00E+000 7 4 300 0 0 00E+000 8 4 700 0 0 00E+000 9 5 100 0 0 00E+000 10 5 500 0 0 00E+000 11 5 500 0 0 00E+000 12 6 300 0 0 00E+000 13 6 700 0 0 00E+000 14 7 100 0 0 00E+000 7 500 0 0 00E+000 7 500 0 0 00E+000

Table C.2 Typical Performance of "Emery 3004" Polyalphaolefin (PAO) in a New TDA-100 Machine; GMD = 0.2083 um, GSD = 1.245.

EMERSOL 875



```
DATE 90/3/9 TIME 110320 PSLF
MAX CNTS/SEC= 758 SEC= 20
P:0-3 TRUN 0 BINS 5.00 ML/SEC
DILUTION RATIO = 1.000E+000
TEMPERATURE (C)= 2.200E+001
ATM PRESSURE (mm H9)= 7.600E+002
REL HUMIDITY (%)= 5.000E+002
REL HUMIDITY (%)= 5.000E+000
B PARAMETER = 0.000E+000
C PARAMETER = 0.000E+000
C PARAMETER = 0.000E+000
C PARAMETER = 0.000E+000
UMD CONC (NUMB/cm3)= 1.560E+002
GEOM HEAN DIRM (vm)= 2.002E-001
GEOM STANDARD UE' = 1.234E+000
MASS CONC (me/m3)= 9.822E-004
UNDIL MASSCON(me/m3)= 9.822E-004
MAX CNTS/SEC = 7.583E+000
FIT NUM CONC (#/cm3)= 0.000E+000
FIT GEOM MN DIA (vm)= 1.000E-008
FIT GEOM STAND DEV = 1.000E+000
FIT GEOM STAND DEV = 1.000E+000
```

PROBE	PANGE=	3 101	CNTS=	13115
BIN	DIA	COUNT	DISTN	
e	120	486		E+001
ĩ	126	398		
÷	132	340	- 65	E+001 E+001
2 3	136	413		E+001
á	144	4-7		E+002
4 5 6 7	150	588		E+003
ž	155	583		E+002
7	162	635		E+002
ģ	169	67.7		E+002
ğ	174	617		E+002
10	189	67.7		E+002
11	195	724	2 28	E+002
12	193	696		E+002
13	198	617		E+002
: 4	204	653		E+002
	210 (4564=0	OVERCOU	NT)

PROBA BIN Ø	E PANGE DIA 170	=2 10T COUNT 6554	CHTS= 15 DISTN WAL 4 03E+0	
i	200 230	4501 2132	3 22E+0 1.74E+0	102 102
234 5	.260 290 720	1016 463 223	9.30E+0 4.70E+0 2.49E+0	101 101
507.00	350 380 410	127 90 57	1 54E+0 1 05E+0 5 24E+0	91
9 10 11	440 470 500	1. 6 1012	5,24E+0 2,88E+0 8,08E-0 3,43E-0	100 101 101
12 13 14	530 560 590	1 0	3.63E-0 1.32E-0 0.00E+0	10 i 100
	620 (4=1	(VERCOUNT	

PROF	BE PANGE=	1 107	CNTS=	773
BIN	DIA	COUNT	DISTN V	
0	300	654	2.27E	
ĭ	400	168	4.84E	
Ž	500	5	2.74E	
2	600	ī	6 49E	
4	700		1.50E	-001
5	300	á	3 00E	+999
6	900	3 3	1.90E	-991
7	1.000	ī	1.05E	-001
8	1.100	Ø	0.00E	+060
9	1 200	0	0.00E	+000
10	1 300	0	0.00E	+000
11	1.400	Э	0 00E	+000
12	1.500	9	0.00E	+998
13	1.600	Э	0 006	+999
14	1.700	9	0 00E	+690
	1.800 (9=0	OVERCOUN	Τ>

PROB	E	RANG	E=0	•	101	CNT	S=		2
BIN		IA		COL	JI-T	DIS	ŤN	Uβ	LUE
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5		500			Ğ				999
ž		988			0	ø			000
7		300			ğ	ø		_	909
7 €		700			ē	ě	a	_	000
ğ		100			ø	Ō		_	000
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ii		900			ğ	ġ	Ã		888
iż		300			ø	ě		_	888
13	2	700			ĕ	ŏ			000
14		100			ě	ě		_	000
. 7		500	(OVER		_	

Table C.3 Typical Performance of Emersol 875 Isostearic Acid 1 a New TDA-100 Machine: GMD = 0.2002 um. GSD = 1.284.

```
DOP
      ANUMCONZAINED: FOIO-24
                                                                                    3
                                                              .170
                                                                         62c7
5054
                                                        0
                                                                                      85E+002
                                                               200
                                                                                    3 62E+002
    IAMETER
                                                                         3103
1579
732
                                                              . 230
                                                                                    2.53E+002
                                                        3
                                                              . 269
                                                                                    1.45E+002
                                                        456789
                                                                                   7.44E+001
3.39E+001
                                                               290
                                                               320
                                                                           364
                                                               350
                                                                           1:8
                                                                                    1.68E+001
                                                               388
                                                                            61
                                                                                    8.03E+000
                                                              .419
                                                                                    1.98E+000
                                                                            14
                                                              .440
                                                                                    1.52E-001
                                                                             1
                                                       10
                                                               478
                                                                                    1 62E-001
                                                       11
12
13
                                                              . 500
                                                                                   0.00E+000
                                                                             0
                                                              539
                                                                                    8.80E+888
                                                              . 569
                                                                                   0 00E+000
                                                       14
                                                              . 599
                                                                             0
                                                                                    0.00E+000
                                                              . 628
                                                                   (
                                                                             1=OVERCOUNT)
                                                       PROBE PANGE=1
                                                                            10T CNTS=
                                                             DIA
                                                                        COUNT
                                                                                 DISTH VALUE
3.50E+001
                                                       BIN
DATE 90/3/9
                   TIME 152420
                                                        Ø
                                                              . 300
                                                                         1008
MAX CHTS/SEC=
                           9EC=
                                     20
                   1116
                                                        1234567
                                                              400
                                                                                   2.82E+000
                                                                            63
R:0-3 TRUN 0 BINS
                           5.00 ML/8EC
                                                              . 500
                                                                                   0.00E+000
DILUTION RATIO
                         = 1.000E+000
= 2.200E+001
= 7.600E+002
                                                              .600
                                                                             9
                                                                                   9 90E+000
TEMPERATURE
                      (C)=
                                                                                     49E-002
ATM PRESSURE
                 (mm Hy)=
                               600E+002
                                                              800
                                                                                   0 00E+000
REL HUMIDITY
                            5.000E+001
                      (%)=
                                                              900
                                                                             ğ
                                                                                   0.00E+000
A PARAMETER
                          =
                            9.000E+000
                                                            1.000
                                                                             0
                                                                                     09E+900
                                                                                   0
  PARAMETER
                          =
                            0.000E+000
                                                        8
                                                            1.100
                                                                             ē
                                                                                   0.00E+000
  PARAMETER
                         =
                            9
                              .000E+000
                                                        9
                                                            1.200
                                                                             0
                                                                                   0.00E+000
NUM CONC
                            2.242E+002
2.004E-001
            (NUMB/cm3)=
                                                       10
                                                                                   8.88E+888
                                                            1.300
                                                                             8
GEOM MEAN DIAM (UM)=
                               884E-881
                                                       11
                                                            1.400
                                                                             ø
                                                                                   0.00E+000
GEOM STANDARD DEL = MASS CONC (M9 m3)=
                               26.2E+888
                                                       12
                                                            1.500
                                                                             0
                                                                                   0.00E+000
                                                       13
                                                            1.600
                                                                             ø
                                                                                   0.00E+000
UNDIL MASSCON(mg/m3)=
MAX CHTS/SEC =
                               210E-003
                                                            1.700
                                                                             0 0.00E+000
0=0VERCOUNT)
                                                                             ø
                               116E+003
                                                            1.800 (
PEAK DIAMETER (PW)
                            1.950E-001
FIT NUM CONC (4/cm3)= 0.000E+000
FIT GEOM MN DIA (PM)=
FIT GEOM STAND DEV =
                                                      PROBE RANGE=0
BIN DIA
                            1.000E-008
                                                                           TOT CHTS=
                         = 1.000E+002
                                                                       COUNT
                                                                                CISTH VALUE
FIT UNDIL MO (mg/m3)= 0.000E+000
                                                        ø
                                                            1 500
                                                                                   0.00E+000
                                                             .900
                                                                                   8 80E+888
                                                            2.300
2.700
                                                        234567
                                                                             Û
                                                                                   0 00E+000
PROBE RANGE=3
                    TOT CNTS= 22314
                                                                                   0.00E+000
      DIA
BIN
                COUNT
                         DISTH VALUE
                                                            3.100
                                                                                   0.00E+000
                   659
      . 120
                            1.35E+002
                                                            3.500
                                                                             9
                                                                                   9 99E+888
                   4.9
      . 126
                            1.07E+002
                                                            3.900
                                                                                   0.00E+000
                            8.66E+001
9 47E+001
      . 132
                   38.5
                                                            4.300
                                                                                   9 99E+999
                   463
547
657
759
      . 138
                                                        89
                                                            4.700
                                                                             ø
                                                                                   0 00E+000
      . 144
                            1.34E+002
                                                              100
                                                                                   0
                                                                                     00E+000
                            1 68E+002
                                                            5.500
                                                      10
                                                                             0
                                                                                   0.00E+000
                            2.01E+002
2.50E+002
3.13E+003
3.13E+002
      . 156
                                                      11
                                                              900
                                                                            ō
                                                                                   8 88E+888
                   968
      . 162
                                                            6
                                                              300
                                                                                   0.00E+000
        168
                  1056
                                                             700
                                                      13
                                                                                   Ø
                                                                                    00E+000
       174
                  1060
                                                             . 100
                                                                            0
                                                                                   0.00E+000
       188
                  1269
1235
                            3.87E+002
                                                            7.500
                                                                             0=OVERCOUNT)
      . 186
                            3 89E+802
      . 192
                            4 23E+002
4 10E+002
                  1361
      . 198
                  1224
1157
       204
210
                            4
                              13E+002
```

Typical Performance of DOP in a New TDA-100 Machine; GMD = 0.2004 um, GSD = 1.262.

(

9113=OVERCOUNT)

0

23456789

10

11

13

Comparison of Performance of DOP, "Emery 3004" PAO, and "Emersol 875" Isostearic Acid in New TDA-100 Machines

Testing was carried out using three new production-line machines at the manufacturer's (ATI's) plant during four visits on February 9, March 9, 12, and April 3, 1990. One material was placed in each machine and allowed to age at temperature, typically 175 °C, for several days. On the following pages, typical data are shown. The geometric mean diameter (GMD) in um and the geometric standard deviation (GSD) are underlined on each chart as printed out by the "LAS-X" laser aerosol spectrometer.

TDA®-100 (Q127) Monodispersed Aerosol Penetrometer

■ The TDA-100 Monodispersed Aerosol Penetrometer (O127) incorporates the most advanced technology of unique design to make .3 micrometer monodispersed aerosol, measure and control the aerosol particle size and concentration plus measure the percent penetration of the tested component by the aerosol.

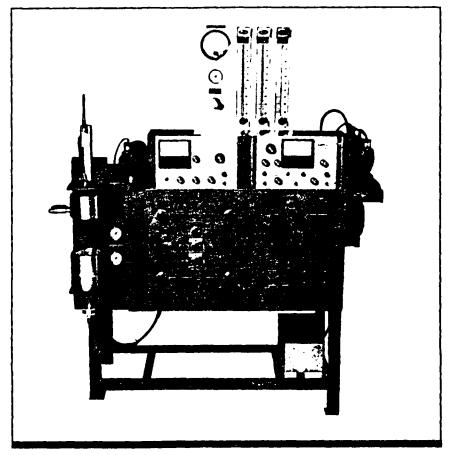
The TDA-100 is a basic apparatus consisting of three major components. They are:

- 1. The penetrometer itself consisting of the aerosol making and controlling equipment.
- 2. The particle size indicator and the mechanical analyzer which monitor the aerosol particle size.
- 3. The percent penetration indicator and associated light scattering chamber which measures the percent of aerosol penetrating the component being tested.

There are many adaptations and possibilities for various chuck and test fixtures which enable testing of a great variety of samples ranging from flat material to highly complex respirators.

In general the TDA-100 operates as follows:

Compressed air, passing through a filter and moisture trap, is connected to the penetrometer and regulated to a pressure of 35 pounds per square inch gage (psig). The air is then divided into two streams, vapor and diluent. The vapor stream flows at 20 liters per minute through a preheater, then into an aerosol generator and over the surface of liquid which is maintained at 165 ± 2°C. The diluent stream is cooled by a vortex tube and then heated by an electrical element. It bypasses the aerosol generator at a flow rate of 80 liters per minute and joins the vapor stream on the outlet side of the generator to make an aerosol. The aerosol is passed into an aging



chamber where it is stabilized. During testing, aerosol flows from the aging chamber to the chuck or test fixture adaptation and through the component under test. As aerosol is continually being made when the penetrometer is operating and testing is intermittent, the excess aerosol is exhausted to the atmosphere from the aging chamber.

The aerosol particle size is maintained at a predetermined level by controls on the penetrometer and is monitored by the aerosol particle size indicator. This indicator electronically measures aerosol particle size from a sample of the aerosol continually passing through a mechanical analyzer. This

mechanical analyzer measures aerosol particle size by the degree of polarization of a light beam which passes through a sample of the aerosol. The particle size of the aerosol is controlled by adjusting the temperature of the diluent air stream.

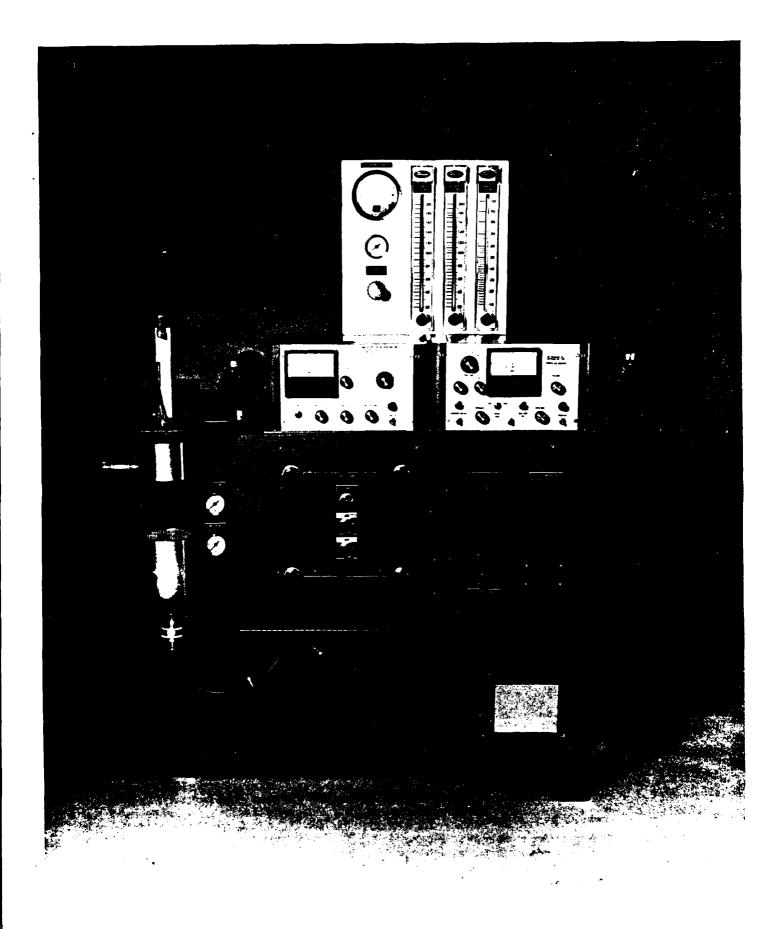
A sample under test is subject to a concentration of aerosol of approximately 100 micrograms per liter. Using this concentration as a base line of 100%, the amount of aerosol penetrating the sample under test is measured by the percent penetration indicator. Such measurements are registered linearly on the meter.



AIR TECHNIQUES

Division of Hamilton Assoc, Inc. 11403 Cronridge Drive Owings Mills, MD 21117-2247 USA

Tel 410 363 9696 Fax 410 363 9695



TDA®-100 Monodispersed Aerosol Penetrometer (Q127)

MAJOR COMPONENTS AND SPECIFICATIONS

- AEROSOL GENERATOR: Produces 0.3 micron aerosol at a concentration of 100 micrograms/liter
- VAPOR FLOWMETER: Ranges from 5-50 SLPM @ 35 PSIG
- DILUENT FLOWMETER: Ranges from 10-100 SLPM @ 35 PSIG
- TEST FLOWMETER: Ranges from 16-85 SLPM @ 5" Hg
- RESISTANCE INDICATOR: Magnehelic Gage. 0-80 MM WATER COLUMN
- MECHANICAL ANALYZER: Measures light-angle polarization from 0 °-50 ° with four polaroid and three condensing lenses
- PARTICLE SIZE INDICATOR: Solid state type, capable sensitivity of ten divisions to 1° rotation of Mechanical Analyzer, approximate size 14″ × 8″ × 8″

- SCATTERING CHAMBER: Forward light scattering, approximately 5" × 5" × 20" in size, with ultra black no smoke feature
- PERCENT PENETRATION METER: Solid state type with ranges of 100%, 10%, 1%, 1%, .01%. Approximate size—14" × 8" × 8". Three place digital read out optional
- VORTEX TUBE: 5 cubic feet per min. capacity
- MIXING CHAMBER: Containing baffles with ports for exhaust, sample, inlet and test sample
- VACUUM PUMP: Capable of delivering up to 85 SLPM @ 5"HG pneumatic, silent operating type
- AIR OPERATED CHUCK: Manufactured to house customers' canisters of varying sizes, etc., to be tested

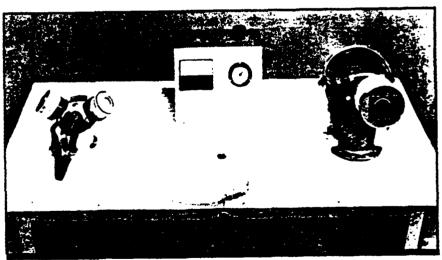
- CONSTANT VOLTAGE REGULATOR: 250 VA rating. Input of 95-130 VAC output of 118 VAC ± 0.5%
- CONTROL PANEL: Consisting of master "ON-OFF" particle size control, solid state time proportioning liquid temperature control, chuck control switches

APPLICABLE STANDARDS AND SPECIFICATIONS

ASTM D 2986-71 Evaluation of Air Assay Media by the Monodisperse DOP (Dioctyl Phthalate)

30 CFR 11 NUREG 0041 MIL STD 282

American National Standards Institute N101.1-1972. ANSI/ASME N510-1980. Institute of Environmental Sciences IES-RP-CC-001-86



A test bench, TDA 101, is available as an accessory to the TDA-100 for testing leaks in facepieces of both full and half mask respirators. The bench has two test heads, a spray nozzle for aerosol, a penetration readout meter, valves, connectors and hardware for hook-up to the TDA-100 Monodispersed Aerosol Penetrometer.

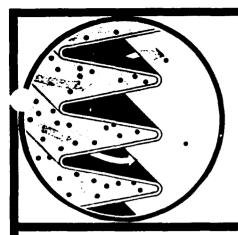


AtR TECHNIQUES
Division of Hamilton Assoc . Inc
11403 Cronridge Drive
Owings Mills, MD 21117-2247 USA
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APPENDIX F

TYPICAL PERFORMANCE DATA:

COLD SMOKE APPLICATIONS



Model 8110 Automated Filter Tester

Features and Specifications

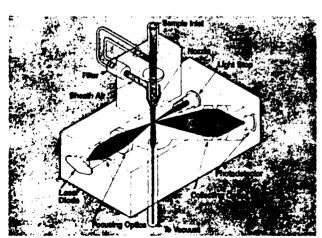
TSI Model 8110 Automated Filter Tester represents a new generation of production filter testers. The TSI tester is faster and easier to operate than presently available production filter testers and requires little operator training. It also uses TSI's solid-state photometer for top notch accuracy over its dynamic range.

The tester generates an aerosol which is used to challenge a test filter. Particle concentrations are measured upstream and downstream of the filter, with filter penetration calculated by a microprocessor. To conduct a test, the operator simply puts a canister or filter in the filter holder and pushes two buttons to close the holder. The test then runs automatically, with the penetration, flow rate and pressure drop displayed at the conclusion.

A microprocessor-based system, the Model 8110 has a self-diagnostic feature which significantly reduces operator training and the need for operator intervention during the test sequence. If a parameter is outside the specified range, the microprocessor declares the test invalid and directs the operator to the source of the problem.

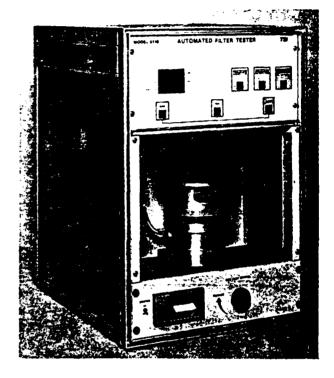
TSI's unique solid-state photometer makes the system highly reliable. The photometer uses a laser diode for the light source and a photodiode as the detector. This combination of solid-state components provides high reliability and stability. TSI's unique off-axis light collection greatly reduces background light levels at the detector. The microprocessor automatically switches gains on the photometer output and corrects for background levels.

Aerosol generators are provided for both salt and oil testing.



The Model 8110 detects particles using a solid-state photometer.

APPENDIX F



Options include the Model 8111 Automated Fit Tester package which converts the Model 8110 to a respirator fit tester for use with any chamber.

Features

- ☐ Fast, reliable filter efficiency measurements
- ☐ Automated operation with minimum operator training
- ☐ Extremely accurate concentration measurements over a wide dynamic range
- ☐ Measures efficiencies to 99.99% and beyond
- ☐ Filter pressure drop and flow rate measurements
- ☐ Self-check diagnostics
- ☐ Easy accessibility in movable cabinet
- ☐ No zero and span adjustments on detector
- ☐ Printer provides hard copy output
- ☐ RS-232 data output
- ☐ Salt and oil aerosol generators

Accessories

- ☐ 102 mm filter holder for concentration verification
- ☐ Orifice plate for flow calibration



Options

■ Model 8111 Automated Fit Tester package

Specifications

Challenge Aerosol

Oil Generator (using DOP)

Technique Atomizer Number Mean Diameter 0.20 micrometer

Geometric Standard Deviation . . . < 1.6

Concentration 15 mg/m³ to 100 mg/m³

Salt Generator (using NaCl)

Technique Atomizer Number Mean Diameter 0.10 micrometer

Geometric Standard Deviation . . . < 1.9 Concentration 15 mg/m³ to 100 mg/m³

Challenge Aerosol Detection

Technique Solid-state photometer

Sample Flow Rate 2.2 1/min

Dynamic Range 1.0 μg/m³ to 200 mg/m³

Accuracy ± 10%

Flow Measurement

Technique Orifice with electronic pressure transducer

Accuracy ± 2% of reading

Range 15 to 110 l/min

Pressure Measurement

Technique Electronic pressure transducer

Accuracy ± 0.5% of full scale Range 0 to 15 cm H₂O

Efficiency Measurements

Flow Rate through Media 15 to 100 l/min
Operating Range Efficiencies to 99.99% and beyond

Automation and

Data Management Dedicated microprocessor system

Operational Requirements

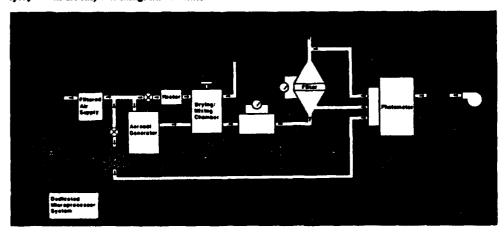
Power 115 VAC, 60 cycle, 4A or 230 VAC, 50 cycle, 2A

Compressed Air 7 cfm @ 80 psi

Physical Characteristics

Weight 210 lb (95 kg)

Specifications are subject to change without notice



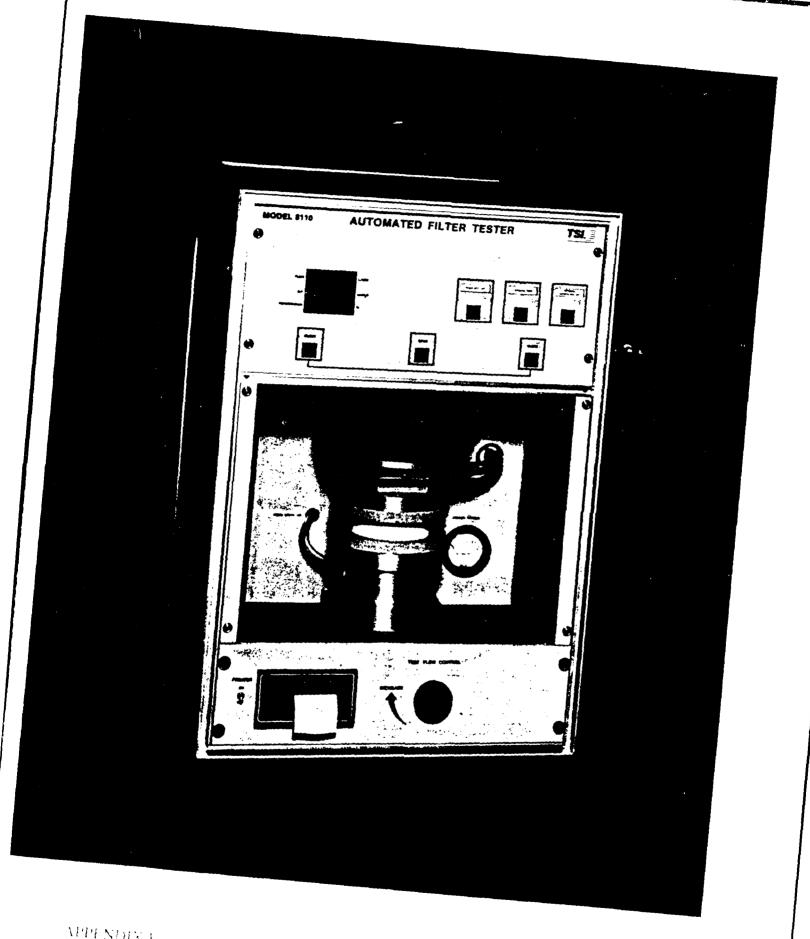


TSI incorporated industrial Test Instruments Group 500 Cardigan Road

PO Box 64394 St. Paul, MN 55164 Telephone: 612 490 2888 Telex: 6879024 Fax: 612 490 2874

TSI GmbH Zieglerstrasse 1 D-5100 Aachen, Germany (F.R.) Telephone: 0241 52303 0 Telex: 83 2219 TSI D Fax: 0241 5230349

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APPENDING E

Testing with the National Institute For Occupational Safety and Health (NIOSH)

APPENDIX I

<u>Table.</u> Mean values of Aerosols generated in the 8110 using candidate materials, in high and low concentration modes.

Material /Mode	Number Dia.	Spread	Surface Dia.	Spread	Volume Dia.	Spread
DOP						
High	0.138	1.528	0.184	1.400	0.203	1.35
Low	0.118	1.705	0.181	1.478	0.206	1.39
Emersol 8	175					
High	0.164	1.512	0.208	1.332	0.223	1.28
High	0.158	1.524	0.203	1.345	0.219	1.29
Low	0.139	1.734	0.201	1.388	0.220	1.31
Low	0.138	1.746	0.200	1.392	0.219	1.31
Emery 300)4					
Low	0.123	1.771	0.192	1.457	0.215	1.35
Low	0.122	1.804	0.194	1.454	0.217	1.34
High	0.158	1.534	0.206	1.356	0.223	1.29
High	0.157	1.53	0.205	1.360	0.222	1.30
Emery 221	.9					
Low	0.054	1.969	0.142	1.838	0.188	1.56
Low	0.052	2.09	0.15	1.828	0.195	1.52
High	0.051	1.853	0.117	1.864	0.162	1.67
High	0.051	1.866	0.115	1.851	0.159	1.66

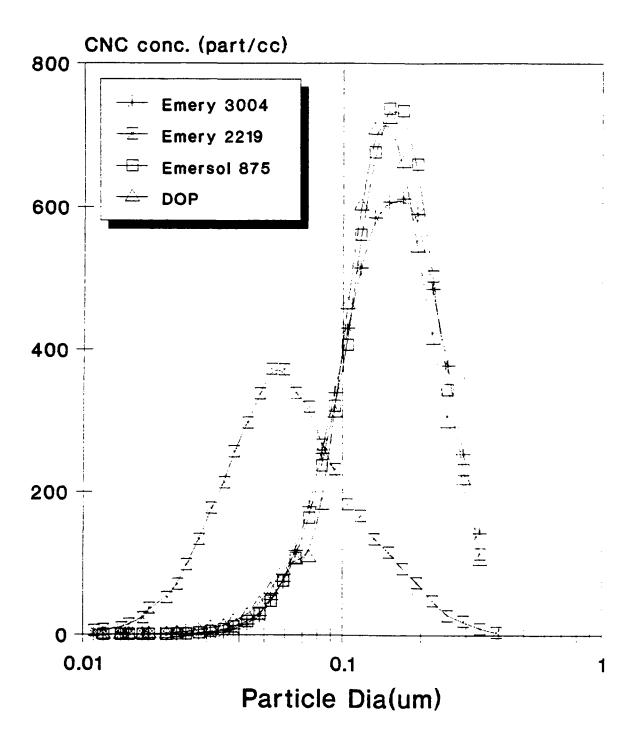
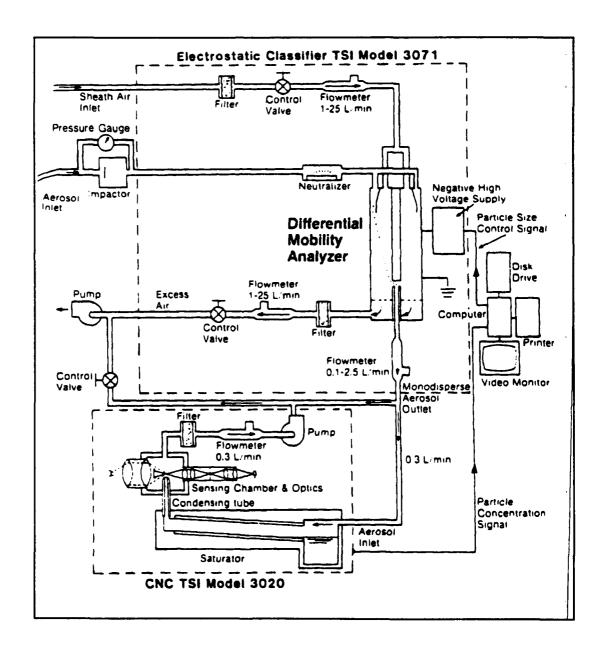


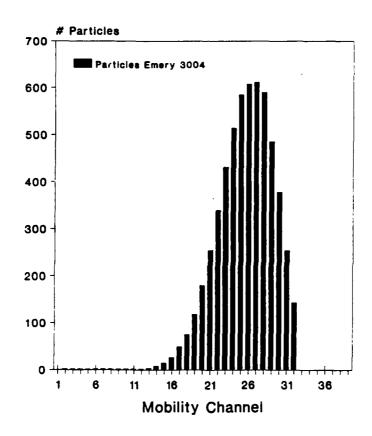
Figure. Schematic view of the DMPS with Electrostatic Classifier (EC) and Condensation Nucleus Counter (CNC). Particles entering the classifier are neutralized prior to being recharged and separated according to their electrostatic mobility. The CNC counts the number of particles in each mobility channel. Data is collected on a microcomputer.



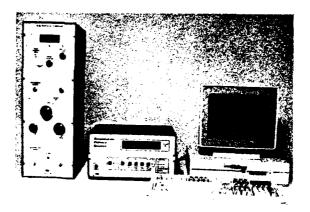
Mass concentrations were calculated for each candidate material in both high and low concentration modes.

DOP	High mode	110.0 ug/L	Low mode	10.0 ug/L
Emersol 875	High mode	114.9 ug/L	Low mode	12.9 ug/L
Emery 3004	High mode	167.0 ug/L	Low mode	16.0 ug/L
Emery 2219	High mode	145.0 ug/L	Low mode	2.0 ug/L

The TSI DMPS prints a chart of particle distribution as particles per mobility channel. Figure is an example of a printout of particle distribution per mobility channel generated with Emery 3004.



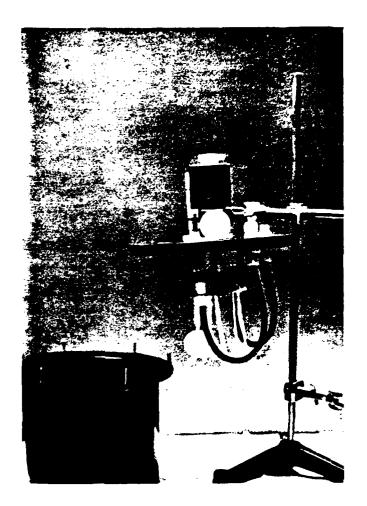
TSI, Inc., DMPS System (Model 3932)



The Model 3932 Differential Mobility Particle Sizing System offers superb resolution in a moderate sampling time for reliable measurement of stable aerosols with submicrometer

particles. Because it resolves sizes over a total of 32 channels spanning the 0.01 to $1.0~\mu m$ range, it allows the operator to observe very slight differences in particle size. The DMPS offers the

unique flexibility of components which, aside from working as integral parts of the system, operate as stand-alone instruments.

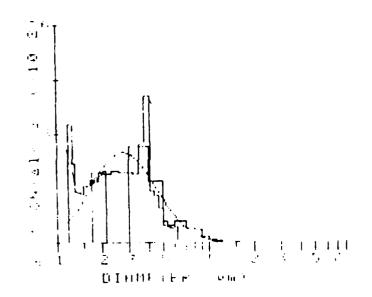


Model 8110 Smoke Generator:

Corn Oil Droplet Distribution from Model 8110 in NIOSH Tests:

Geometric Nean Diameter= 0.2744 um (GND)

Geometric Standard Deviation= 1.647 (GSD)



Consultative Assistance to Los Alamos National Laboratory (LANL)

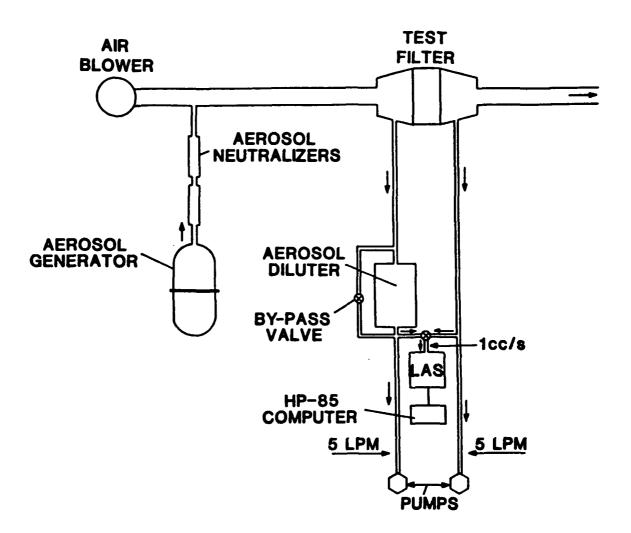


Fig. J-1. A schematic diagram of the ATS showing the major components of the system, which includes the aerosol generator, the aerosol neutralizers, the ATS aerosol diluter, the laser aerosol spectrometer, and the HP-85 computer.

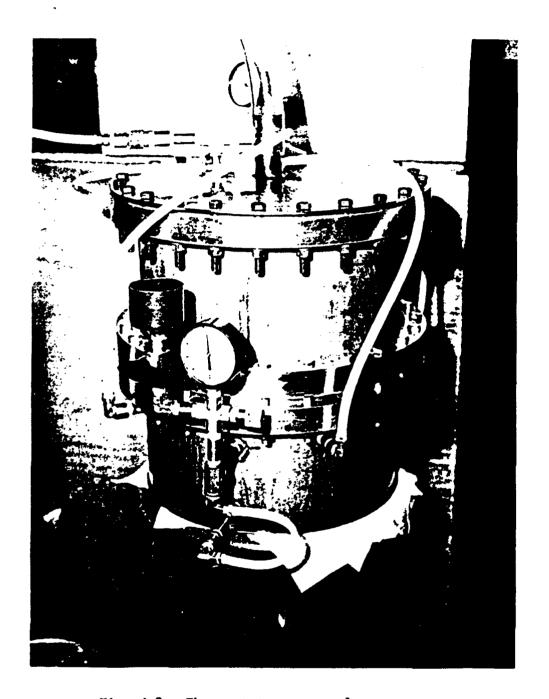


Fig. J-2. The prototype aerosol generator.

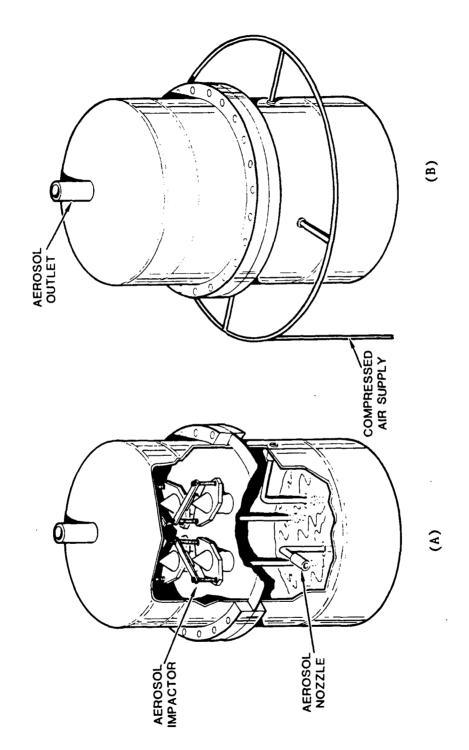
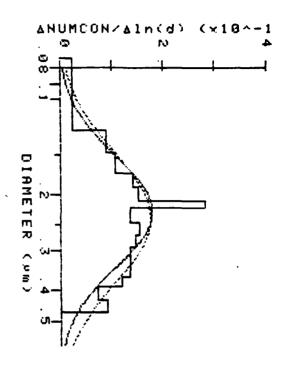


Fig. J-3. A diagram with two views of the ATS aerosol generator. View A shows the internal components of the generator and View B shows the compressed-air manifold.

CURRENT OFTIONS:
PENBIN IS # 9
DILRAT FOR PENBIN IS 248.4
PRINT PENBIN PENETRATION ONLY.

100%

UPSTREAM: 1887 CNTS/SEC



Material: Emery 3004

Flow: 1500 CFM

Pressure: 15 PSI

Generator(s): Two

Fig. J-5. HFATS Data (See Report Section 2.3.2.1)

DATE 90/10/23 TIME 82636 DEHP 1.00 (mL/SEC) 100010 SEC DILUTION RATIO = 1.000E+000 DIL TOT FLOW (L/min) = 0.000E+000 DIL ORIF AP (IN H20)= 0.000E+000 ONL POL. ANGLE (DEG)= 0.000E+000 **TEMPERATURE** (C) = 0.000E + 000ATM PRESSURE (mm Hg)= 0.000E+008 REL HUMIDITY (%) = 0.000E + 000HUM CONC (NUMB/cm3) = 1.813E-001GEOM MEAN DIAM (Jm)= 2.264E-001 GEOM STANDARD DEV = 1.493E+600(m9/m3) = 2.014E-006MASS CONC UNDIL MASSCON(mg/m3)= 2.014E-006 MAX CNTS/SEC = 1.813E-001PEAK DIAMETER (Pm) = 2.144E-001 FIT NUM CONC (#/cm3)= 2.093E-001 FIT GEOM MN DIA (um)= 2.366E-001 FIT GEOM STAND DEV = 1.602E+000 FIT UNDIL MO (mg/m3)= 3.946E-006

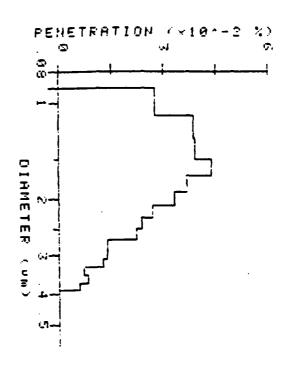
BIN DIA COUNT DISTN VALUE 8 . 075 1243 2.40E-002 .126 1 1461 9.08E-002 1551 234 . 148 1.07E-001 1.43E-001 1.52E-001 .171 1436 1527 . 189 2.83E-001 1.37E-001 567 . 209 1452 1471 . 220 . 245 1.57E-001 1346 8 1276 . 267 1.48E-001 9 1172 1.37E-001 . 291 19. 1.38E-001 .317 1005 11 .341 862 1 38E-001 12. . 363 784 1.22E-00i

- }

CURRENT OPTIONS: PENBIN IS # 9 DILUTION RATIOS ARE: BIN # 246 244 245 DILRAT 248 8 BIH # 245 261 242 233 248 DILRAT 12 255 13 10 11 14 BIN # 240 248 258 248 DILRAT PRINT ALL PENETRATION VALUES

100% UPSTREAM: 1486 CNTS/SEC DOWNSTREAM: 101 CNTS/SEC

FILTER ID: 1500 CFM BIN 9 PENETRATION = 013 %



Percent Penetration Data:

Material:

Emery 3004

Flow:

٠.

1500 CFM

Percent of

Filter Capacity: 100%

Fig. J-6. HFATS Data (See Report Section 2.3.2.1)

PENETRATION TABLE
(CALCULATED ONLY FOR BINS WITH 10 PARTICLES OR MORE)

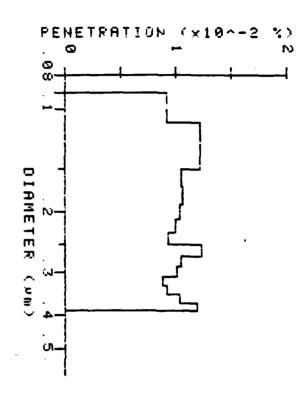
79 0	B 0 1 2 3 4 5 6 7 8 9 0 1 1 2 3 1 4	DIAM 09100000000000000000000000000000000000	PEN (%) 2.75E-002 3.88E-002 4.38E-002 3.67E-002 2.68E-002 2.37E-002 2.23E-002 1.38E-002 1.38E-002 1.28E-003 1.28E-003 5.14E-003
		390	

20%

UPSTREAM: 1506 CHTS/SEC DOWNSTREAM: 39 CNTS/SEC

FILTER ID:

1500 CFM BIN 9 PENETRATION = .01 %



PENETRATION TABLE (CALCULATED ONLY FOR BINS WITH 10 PARTICLES OR MORE)

BIN	DIAM.	PEN (%)
Ø	.090	9.258-003
1	. 110	1.226-002
2	. 130	1.22E-002
3	. 150	1.05E-002
4	.170	1.06E-002
5	. 190	1.04E-002
6	. 210	1.00E-002
7	. 230	9.31E-003
8	. 250	1.23E-002
9	.270	1.05E-002
10	. 290	1.025-002
11	. 310	8.78E-003
12	. 330	9.27E-003
13	. 350	1.04E-002
14	. 370	1.20E-002
	. 390	

Percent Penetration Data:

Emery 3004 Material:

1500 CFM Flow:

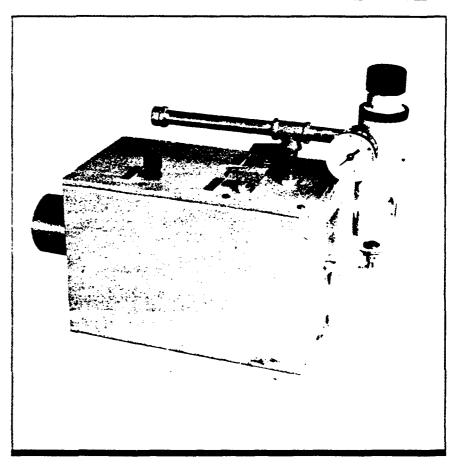
Percent of

Filter Capacity: 20%

Fig. J-7. HFATS Data (See Report Section 2.3.2.1)

TDA®-4A Aerosol Generator

The TDA-4A Aerosol Generator is used to detect leaks in air filtration systems. It is simple and easy to operate requiring only liquid aerosol and dean, compressed air. Based on the Laskin nozzle type apparatus, it creates a liquid aerosol of consistent particle size distribution by shearing the liquid with air. Used in conjunction with the TDA Particulate Detection Units, it will quickly and efficiently test clean benches, filter banks, clean rooms and filter units for leaks. The controls allow fractions of the entire aerosol output to be used to compensate for different conditions. When the entire output is diluted by 1,000 cfm of air, the aerosol concentration is approximately 100 micrograms per liter. With both valves closed, one nozzle is in operation and combinations of three, six, and eight nozzles may be obtained by opening or closing the two valves. With increased sensitivity of the detection units it is possible to test for leaks in filter banks up to 4,000 cfm, but the operator is cautioned that a test performed in this fashion will be done at less than 100 micrograms per liter aerosol concentration.



APPLICATIONS

This equipment is applicable for industrial, medical, pharmaceutical and other types of contamination control installations . . . for monitoring, controlling, or leak testing the following:

- Filters—any size or efficiency . . . especially for pinhole leaks at lower than rated flow for high efficiency units.
- Work Stations—any work station using filters . . . for leaks around seals, gaskets, filter holders, and any other possible sources of leaks.
- Clean Rooms filter systems of laminar or non-laminar rooms, tunnels, or similar units . . . for leaks around seals, gaskets, or filter frames.
- Filter Banks filter systems of laminar or non-laminar flows, for influent or effluent air handling . . . for leaks around seals, gaskets, or filter frames.



TDA®-4A Aerosol Generator

CONSTRUCTION

Aluminum

SIZE 12" × 7" × 8"

■ WEIGHT

15 lbs.

■ PARTICLE SIZE DISTRIBUTION

99% + less than 3.0 micron 95% less than 1.5 micron

92% less than 1.0 micron

50% less than 0.72 micron

25% less than 0.45 micron

10% less than 0.35 micron

CAPACITY

Up to 4,000 cfm Systems

UTILITIES

24 cfm clean air at 20 psi

OPERATING TEMPERATURE

Ambient

APPLICABLE SPECIFICATIONS AND STANDARDS

U.S. Federal Standard 209b, paragraph 50.

American Association of Contamination Control standards CS-1T, CS-2T and CS-6T.

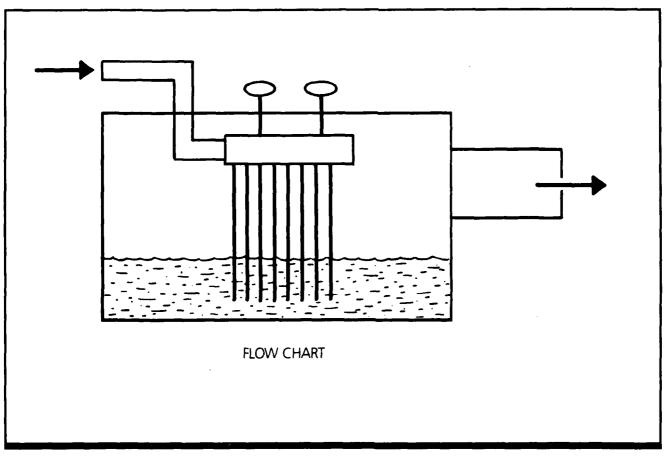
American National Standards Institute N101.1-1972.

ANSI/ ASME N510-1980.

National Sanitation Foundation No. 49.

Institute of Environmental Sciences IES-RP-CC-001-86

Institute of Environmental Sciences IES-RP-CC-002-86



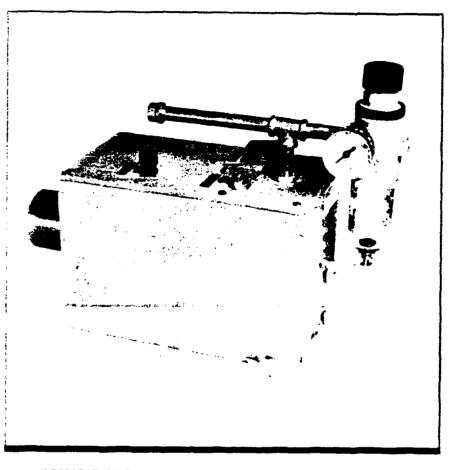




AIR TECHNIQUES DIVISION OF HAMILTON ASSOCIATES INC. 71716 WHITEHEAD ROAD BALTIMORE, MARYLAND 21207 TELEPHONE (301) 944-6037 FAX (301) 298-3617

TDA®-4A Aerosol Generator

■ The TDA-4A Aerosol Generator is used to detect leaks in air filtration. systems. It is simple and easy to operate requiring only liquid aerosol and clean, compressed air. Based on the Laskin nozzle type apparatus, it creates a liquid aerosol of consistent particle size distribution by shearing the liquid with air. Used in conjunction with the TDA Particulate Detection Units, it will quickly and efficiently test clean benches, filter banks, clean rooms and filter units for leaks. The controls allow fractions of the entire aerosol output to be used to compensate for different conditions. When the entire output is diluted by 1,000 cfm of air, the aerosol concentration is approximately 100 micrograms per liter. With both valves closed, one nozzle is in operation and combinations of three, six, and eight nozzles may be obtained by opening or closing the two valves. With increased sensitivity of the detection units it is possible to test for leaks in filter banks up to 4,000 cfm, but the operator is cautioned that a test performed in this fashion will be done at less than 100 micrograms per liter aerosol concentration.



APPLICATIONS

This equipment is applicable for industrial, medical, pharmaceutical and other types of contamination control installations . . . for monitoring, controlling, or leak testing the following:

- Filters—any size or efficiency . . . especially for pinhole leaks at lower than rated flow for high efficiency units.
- Work Stations—any work station using filters . . . for leaks around seals, gaskets, filter holders, and any other possible sources of leaks.
- Clean Rooms filter systems of laminar or non-laminar rooms, tunnels, or similar units . . . for leaks around seals, gaskets, or filter frames.
- Filter Banks—filter systems of laminar or non-laminar flows, for influent or effluent air handling . . . for leaks around seals, gaskets, or filter frames.



Blank

APPENDIX G

WESTINGHOUSE HANFORD COMPANY
TECHNICAL REPORT

Emery 3004 as a Challenge Aerosol: Operational Experience at Westinghouse Hanford Company

Prepared for the U.S. Department of Energy Office of Environmental Restoration and Waste Management



Hanford Operations and Engineering Contractor for the U.S. Department of Energy under Contract DE-AC06-87RL10930

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APPENDIX G

Emery 3004 as a Challenge Aerosol: Operational Experience at Westinghouse Hanford Company

D. H. Steffen C. K. Girres

Date Published October 1992

Prepared for the U.S. Department of Energy Office of Environmental Restoration and Waste Management



Hanford Company P.O. Box 1970
Richland, Washington 99352

Hanford Operations and Engineering Contractor for the U.S. Department of Energy under Contract DE-AC06-87RL10930

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EMERY 3004* AS A CHALLENGE AEROSOL: OPERATIONAL EXPERIENCE AT WESTINGHOUSE HANFORD COMPANY

D. H. Steffen C. K. Girres

ABSTRACT

High Efficiency Particulate Air (HEPA) filter systems are tested periodically by chemicals such as dioctyl phthalate (DOP) and di-2-ethylhexyl sebacate (DOS) to ensure adequate performance. For eight months, Westinghouse Hanford Company used Emery 3004 as a challenge aerosol for in-place HEPA filter system testing.

Operationally, Emery 3004 has several advantages over approved performance testing chemicals, including that it is not considered a carcinogen or suspect carcinogen; therefore, respiratory protection is not required during testing. Additionally, Emery 3004 does not cause buildup on or plugging of the test equipment like DOP or DOS. By reducing the maintenance required on equipment, use of Emery 3004 increases the efficiency of Westinghouse Hanford Company operations.

The concern with using Emery 3004 for in-place testing of HEPA filter systems has been the lack of definitive data on its particle size distribution when generated with a "cold smoke" generator. Quantitative data was not available to show compliance with the particle size distribution requirements of American National Standards Institute, American Society of Mechanical Engineers N510, the standard for in-place testing. To provide comparative

^{*}Emery 3004 is a trademark of the Henkel Corporation. APPENDIX G

data between DOP, DOS, and Emery 3004, Westinghouse Hanford Company performed a practical field demonstration, and the results indicated that Emery 3004 behaved like the traditional aerosols. Additional preliminary tests were conducted to obtain particle distribution data, and, as a result of this testing, Westinghouse Hanford Company has received approval from the U.S. Department of Energy-Headquarters, Office of Engineering and Operations Support Defense Programs, and the U.S. Department of Energy, Richland Field Office, to use Emery-3004 as a challenge aerosol agent for the in-place testing of high-efficiency particulate air filter systems via U.S. Department of Energy letter from J. R. Hunter, Assistant Manager for Operations to T. M. Anderson, President of Westinghouse Hanford Company, dated September 24, 1992.

This paper discusses the operational advantages of Emery 3004 and further discusses our test results.

EMERY 3004 AS A CHALLENGE AEROSOL: OPERATIONAL EXPERIENCE AT WESTINGHOUSE HANFORD COMPANY

Several studies have taken place to look for noncarcinogenic replacement aerosols that behave similarly to dioctyl phthalate (DOP). These studies, which analyze materials that are not carcinogens or suspected carcinogens, are based on efficiency testing.

One of the most recognized studies nationally is A Study of Candidate Replacement Materials for DOP in Filter-Testing Penetrometer Machines (Carlon and Guelta 1989). This study was performed by the U.S. Army Armament Chemical Command at Aberdeen Proving Ground, Maryland, and focused on identifying viable candidates to replace DOP as a challenge aerosol. Emery 3004* was chosen as one of the candidate materials.

Both a cold smoke machine and a hot smoke machine similar to the one used in the field comparison testing were used to obtain data. The accepted U.S. Army standard for hot smokes is a geometric mean diameter (GMD) of 0.3 μ m, a geometric standard deviation (GSD) equal to or less than 1.3, and a mass concentration of 100 mg/m³. The experimental procedures were based on these specifications. The hot smoke machine used is known as a Q127, model number TDA-100** manufactured by Air Techniques Incorporated (ATI). The particle size was monitored with a laser aerosol spectrometer.

The approach used to test the candidate materials was to look at the properties of DOP that make it desirable as a test aerosol. DOP is characterized by its low vapor pressure, chemical stability, and insolubility in water. Material properties that were considered include reproducible particle size, size distribution, and smoke concentration (a function of the material density and particle diameter).

The penetrometer used was adjusted to achieve the appropriate particle size and distribution. The GMD was allowed to vary from $0.2\mu m$ to $0.3\mu m$ because recent recommendations for the penetrometer operations include the use of particles smaller than $0.3\mu m$ because they are more effective in penetrating modern filters than are larger particles. To maintain the same vapor pressure, the temperature of the pot was varied.

When Emery 3004 was tested, the GMD was adjustable from $0.2\mu m$ to $0.3\mu m$, a GSD of 1.23 was obtained, and it had an adequate aerosol yield. The test results showed that Emery 3004 has a high potential as a replacement material for DOP/di-2-ethylhexyl sebacate (DOS). Sample test results for DOP, DOS, and Emery 3004 from the report are shown in Table 1 (Carlon and Guelta 1989).

^{*}Emery 3004 is a trademark of the Henkel Corporation.

^{**}TDA-100 is a trademark of Air Techniques Incorporated.

i	GMD (μm)	GSD	Pot Temperature (°C)
DOP	.2581	1.542	172
DOS	.3017	1.161	185
Emery 3004	.2944	1.230	180

Table 1. Sample Test Results for COP, DOS and Emery 3004.

The report concludes by recommending Emery 3004 synthetic hydrocarbon as one of two probable replacement materials and recommends further testing to ensure adequate stability and aging characteristics as well as initiating toxicological screening.

A followup of this study was presented at the 21st DOE/NRC Nuclear Air Cleaning Conference: Safe Replacement Materials for DOP in "Hot Smoke" Aerosol Penetrometer Machines (Carlon and Guelta 1991). The conclusion of this paper was that a synthetic hydrocarbon can be used to replace DOP directly with minimum impact upon existing hardware and procedures.

In addition to the work described in the 1989 study, further testing was performed to determine the effects of aging and temperature on the stability of Emery 3004. Testing showed that Emery 3004 was sufficiently similar to DOP when subjected to evaporation and recondensation in filter penetrometer testers. The next step in the experimentation process showed that Emery 3004 is thermally stable when subjected to aging tests at elevated temperatures. DOP and Emery 3004 were observed to have similar aging properties.

Westinghouse Hanford Company used Emery 3004 as a challenge aerosol for in-place penetration testing of high-efficiency particulate air (HEPA) filters from May 2, 1991, through January 7, 1992. During this time period, 427 HEPA filter systems were tested. The test results were compared to earlier and subsequent tests that used DOS as the challenge agent on the same filter systems. The results of the tests indicated that the readings gathered from the filter systems using Emery 3004 were virtually identical to those using DOS as the challenge agent.

Additional challenge aerosol comparison tests were conducted at Westinghouse Hanford Company to compare the performance of three challenge aerosols: DOP, DOS, and Emery 3004. The tests were conducted using equipment that is normally used for testing HEPA filter systems in the field at the Hanford Site. Actual field conditions were simulated in the Vent and Balance Laboratory in the 2101-M building in the 200 East Area.

The comparison tests were conducted on January 9, 1992. Three ATI Model TDA-5A* smoke generators were used, one for each aerosol. Each smoke generator was set to the test equipment and aerosol manufacturers' specifications. Three smoke generators were used to facilitate performance of

^{*}TDA-5A is a trademark of Air Techniques Incorporated.

the tests in as short a time span as possible to avoid fluctuations in ambient air pressure and temperature. An ATI Model TDA-2E penetrometer was used to measure smoke penetration through the test filters.

Two 61 x 61 x 30.5 cm (24 x 24 x 12 in.) HEPA filters were used for the tests. The first test series was conducted using an old filter that had been used for several years. The second test series was conducted using a new filter. The third test series was conducted using the old filter with an intentional perforation of 0.16 cm (1/16 in.) diameter located at the filter center.

These three tests were performed to generate data that was as similar as possible to actual field conditions and provide a realistic comparison of the aerosols. All test data, conditions, filters, instrument calibrations and test methods were witnessed and verified by Hanford Site quality control and industrial safety personnel.

The DOP aerosol was tested at the beginning and conclusion of each series of tests to "bracket" the other two aerosols and ensure that the test conditions remained constant. The data indicates that Emery 3004 performs almost identically to DOP and slightly better than DOS. All three aerosols performed within 0.15 percent of each other.

The test data was analyzed only by comparing data within each individual test set (Table 2). Comparisons of data were not made between test sets because the test conditions can change because of factors such as how much air leakage occurs around the filter installation. The purpose of the test was to look at aerosol performance under field conditions, so no effort was made to standardize these conditions.

In the two tests performed on the old filter, it failed in all cases. In addition, for the new filter, the minimum specification was met in all cases. All of the data was within 0.002 percent of each other, which is the standard allowed for the subjectivity in reading the penetrometer. No unusual observations were made that would invalidate the data.

INTEGRATION OF ANALYSES

Several conclusions can be drawn by looking at our field data and comparing it to laboratory experiments. The U.S. Army literature indicates the critical factor when looking for an equivalent aerosol is the particle distribution. The material chosen must exhibit chemical properties similar to DOP to meet the specifications for both the aerosol and the test machine.

The primary difference between the Hanford Site field test and the tests done at Aberdeen Proving Ground is in the test process itself. The Aberdeen Proving Ground test was designed for efficiency testing. For these tests, an aerosol having a monodispersed particle size is specified. The in-place leak tests done in the field are based on a polydispersed particle size meeting the requirement of American National Standards Institute/American Society of Mechanical Engineers N510 (ANSI/ASME 1989b).

Several similarities between the two tests allow for favorable comparison of the results and the ability to draw accurate conclusions. First, although the smoke generators used were different (as discussed above), they are both manufactured by the same company and operate on the principle of a hot pot to generate the aerosol smoke. The 21st DOE/NRC Air Cleaning Conference Abstract states that the data obtained should be applicable to all hot pot machines (Carlon and Guelta 1991). Secondly, the Aberdeen Proving Ground tests were based on finding replacements based on chemical similarities and toxicological data. The data discussed in this paper supports the conclusion that Emery 3004 is an effective challenge aerosol and viable substitute for both DOP and DOS.

Table 2. Challenge Aerosol Comparison Test Data.

Test Number 1 (Used HEPA filter)					
Aerosol	Range	Penetration	Reading		
DOP	1%	0.6%	99.4%		
DOS	1%	0.7%	99.3%		
Emery 3004	1%	0.6%	99.4%		
DOP	1%	0.7%	99.3%		

Test Number 2 (New HEPA filter)					
Aerosol	Range	Penetration	Reading		
DOP	1%	0.002%	99.998%		
DOS	1%	0.002%	99.998%		
Emery 3004	1%	0.003%	99.997%		
DOP	1%	0.004%	99.996%		

Test Number 3 (Used HEPA firter, perforated)					
Aerosol	Range	Penetration	Reading		
DOP	1%	0.3%	99.7%		
DOS	1%	0.2%	99.8%		
Emery 3004	1%	0.35%	99.65%		
DOP	1%	0.3%	99.7%		

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